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FINAL REPORT
US EPA EXTRACTION METHOD DEVELOPMENT STUDY FOR TRACE METALS IN LEACHATE

by

ERCO/A Division of ENSECO
205 Alewife Brook Parkway
Cambridge, Massachusetts 02140

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A study was performed to determine the applicability of currently approved analytical methods for conducting the EP toxicity test required by the Hazardous Waste and Consolidated Permit Program. The first phase of the study was designed to determine the necessity for performing digestion of the EP leachates prior to trace element analysis and also to determine the effect, if any, of preservation on total metal concentrations. The second phase of the study was designed to determine the effect of pH and time on metal concentrations in the leachate from low-pH sludge. The third phase of the study was designed to determine the efficiency of the EP toxicity procedure in extracting metal spikes from three standardized reference materials. A study was also performed to determine if metal concentrations were affected by various matrices encountered using the EP procedures (acetic acid [0.6%], nitric acid [0.5%], or acetic acid [0.6%]/nitric acid [0.5%]).

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati, conducts research to:

- o Develop and evaluate methods to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
- o Investigate methods for the concentration, recovery, and identification of viruses, bacteria and other microbiological organisms in water; and, to determine the responses of aquatic organisms to water quality.
- o Develop and operate an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.
- o Develop and operate a computerized system for instrument automation leading to improved data collection, analysis, and quality control.

This publication reports the results of a study to evaluate the EP toxicity test.

Federal agencies, states, municipalities, universities, private laboratories and industry should find this study of assistance in monitoring trace metal pollution in the environment.

Robert L. Booth
Director, EMSL-Cincinnati

ABSTRACT

A study was performed to determine the applicability of currently approved analytical methods for conducting the EP toxicity test required by the Hazardous Waste and Consolidated Permit Program. The first phase of the study was designed to determine the necessity for performing digestion of the EP leachates prior to trace element analysis and also to determine the effect, if any, of preservation on total metal concentrations. Four matrices were used for this phase of the study (river sediment, fly ash, low-pH sludge, and oil/water waste) and analyses were performed for 17 elements by both atomic absorption spectrophotometry (AAS) and by Inductively Coupled Argon Plasma Emission Spectrometry (ICP). Digestion of the leachates generally reduced interferences and improved the accuracy of the analyses. For one high sulfur waste, however, digestion introduced a chemical interference. Acid preservation of the wastes did not significantly affect the results.

The second phase of the study was designed to determine the effect of pH and time on metal concentrations in the leachate from low-pH sludge. Determinations were made for 17 elements in the EP extracts after adjustment of initial pH to levels of 2, 4, 6, and 8. Extractions were performed 0, 2, 8, and 16 hours following pH adjustment. All wastes exhibited a strong dependence on the initial pH of the wastes - the leachate concentrations were lowered at higher initial pH values. The time between pH adjustment and initiation of the extraction procedure did not alter the results.

The third phase of the study was designed to determine the efficiency of the EP toxicity procedure in extracting metal spikes from three standardized reference materials (river sediment, fly ash, and EPA Municipal Digested Sludge). The results of this phase confirmed the strong pH dependence of leachate concentrations found in Phase 2 and demonstrated that under normal EP extraction conditions extraction efficiencies for most metals are low.

A study was also performed to determine if metal concentrations were affected by various matrices encountered using the EP procedures (acetic acid [0.6%], nitric acid [0.5%], or acetic acid [0.6%]/nitric acid [0.5%]). Of all the 17 elements studied, only selenium exhibited a matrix effect from the dilute acids. The slopes of the selenium calibration curves generated by both ICP and AAS were enhanced in the presence of acetic acid.

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SECTION 1

INTRODUCTION

The Hazardous Waste and Consolidated Permit Regulations as published in the Federal Register[1] require testing of wastes to determine their mobility in the soil. This test is termed the EP toxicity test and is designed to test the leachability of toxic wastes and their subsequent transfer to groundwater.

This investigation was conducted in three separate phases with each phase focusing on specific questions related to the applicability of various aspects of approved methods for conducting the EP toxicity tests and subsequent analyses. Phase 1 of the study was designed to determine the necessity of performing leachate digestion after the EP toxicity test and to determine the effect of preservation on metal concentrations. Preservation of the materials used in the study did not affect results. Digestion of the leachates generally reduced interferences and improved accuracy. Four materials were tested - river sediment, fly ash, a low-pH sludge, and an oil/water waste. For the oil/water waste, a separate extraction and analysis was performed on the oil phase. For all extractions, analyses were performed in triplicate using atomic absorption spectrophotometry (AAS) and inductively coupled argon plasma emission spectroscopy (ICP).

The second phase was designed to determine the effect of pH and time on leachate metal concentrations from the low-pH sludge. The waste was extracted at 0, 2, 8, and 16 hr after pH adjustment, with the pH adjustments being made at levels of 2, 4, 6, and 8 with 1.0 M sodium hydroxide. Analyses were performed in triplicate using both AAS and ICP. The results showed a strong pH effect but no time dependence for the leachate concentrations.

The third phase of the study was designed to determine recovery of metal spikes from three standardized reference materials. The three standardized reference materials (river sediment, fly ash, and EPA Municipal Digested Sludge) were spiked in triplicate at three levels, extracted according to the EP toxicity test procedures, and analyzed using AAS and ICP. Recoveries were consistently low, were pH dependent and indicated that the EP procedure measures only the most mobile (or labile) of metals present in a waste.

Preliminary to all three phases, a calibration study was performed to determine the effect, if any, of the acid matrix (0.6% acetic, 0.5% nitric or 0.6% acetic and 0.5% nitric) on the slopes of the calibration curves. Only selenium exhibited a matrix effect (an enhancement in the presence of acetic acid.)

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

The EP toxicity test was designed to indicate the concentrations of eight metals (As, Ag, Ba, Cd, Cr, Hg, Pb, and Se) present in a waste in forms which would allow them to become mobile (solubilized) in a mildly acidic (pH 5 acetic acid) medium. Analyses of the generated leachates are performed by atomic absorption spectrophotometry (AAS). The complete EP procedure is appended to this report (Appendix J).

The purpose of this study was to provide further information regarding various aspects of the EP toxicity test.

Data were generated which were used to address the following:

- Whether an alternative analytical finish (ICP rather than AAS) would provide comparable data.

- Whether the dilute acids encountered in the procedure cause matrix interferences.

- Whether the procedure is applicable to more elements than the original eight.

- Whether sample preservation techniques or sample holding times are significant.

- Whether acid digestion of the generated leachates is necessary.

- Whether the extraction efficiency of the EP procedure is near 100% or whether the "mobile" metal concentrations are significantly less than the total concentrations present.

The conclusions which may be drawn from this study and recommendations based upon the conclusions are summarized below.

CALIBRATION STUDY

- For 16 of the 17 metals studied, the dilute nitric acid or dilute acetic acid matrices used for the EP toxicity test had no effect on the slopes of the calibration curves. The acetic acid matrix enhanced the Se signal by both AAS and ICP. Since enhancements of almost 10% were found for both AAS and ICP, it is recommended that the method of standard additions be used for Se analyses or at least that a test be performed to insure that standard additions are not needed for every EP leachate.

PHASE 1 (Acid Preservation and Digestion Study)

- Digestion of the EP leachates removes several interferences and is generally recommended prior to analysis by either AAS or ICP. However, digestion may cause losses from certain samples such as the high sulfur containing fly ash studied here. Digestion caused formation of sulfate and subsequent precipitation of Ba, Ag, and Pb.

- Acid preservation of the samples used in this study caused little difference in analytical results.

- The ICP analyses of undigested leachates were less subject to interference than were AAS analyses.

- Even though the leachate digestion of the fly ash resulted in losses of Ba, Ag, and Pb for this sample, it is recommended that the digestion be performed. Several matrix interferences in all the wastes were eliminated by the digestion procedure which otherwise would have required time consuming techniques such as standard additions.

PHASE 2 (pH and Storage Time Study)

o The time that a sample is stored (0 to 16 hours) prior to the initiation of the EP leachate generation had no effect on the metal concentrations in the leachate.

o The pH of the sample prior to the initiation of the EP leachate generation is extremely important. This may be true only for selected wastes such as the ones used in this study, i.e., one with a high Fe content which may precipitate at pH 5 or one with a high sulfate concentration which may also cause precipitation. It would be useful to perform Phase 2 on wastes where hydroxide or sulfate precipitation were less likely.

The fact that the leachate concentrations are pH dependent was further confirmed by the Phase 3 data. Due to this variability in recovery (extraction efficiency) it is recommended that the intent of the EP toxicity test be emphasized i.e., that it is an indicator of the concentration of mobile metal species not a total extraction procedure.

PHASE 3 (Extraction Efficiency Study)

o The pH 5 dilute acetic acid matrix used for the leachate generation in the EP toxicity test is not capable of leaching many metals from a solid waste and is incapable of preventing precitation or adsorption of metal ions spiked into the solution. In fact, a river sediment containing 5,700 ug/g Al and 9,200 ug/g Fe was leached with an acetic acid pH 5 solution containing 42.5 mg/L Al (and 68.5 mg/L Fe) with recoveries of the metals in solution being less than 3%.

- As in Phase 1, the ICP analysis of undigested leachates was less subject to interference (as measured by % recoveries) than was AAS.

SECTION 3

METHODS AND MATERIALS

This section summarizes methods for sample preparation for each phase of the study. Instrumental methods are summarized in Table 1.

SAMPLE PREPARATION

Calibration Study

Standards for the calibration study were prepared by dilution of 1000 ppm Fisher or Ventron certified reference standards with the appropriate matrix. Ultrex acetic and nitric acids were used for preparation of the standard matrices (0.6% acetic acid, 0.5% nitric acid, and 0.6% acetic acid/0.5% nitric acid). Analytical wavelengths and the concentrations of standards used throughout the study are shown in Table 1. Mixed standards were used for all elements except As, Hg, Se and Ag which were prepared individually and were prepared daily to minimize losses. Additives and matrix modifiers for AAS analyses are noted as remarks in Table 1.

Flame Atomic Absorption analyses were performed by aspirating each standard and integrating the absorbance for 5 sec with the instrument microprocessor. The instrument used was a Perkin Elmer Model 603. A distilled, deionized water rinse was used between standard aspirations. Each standard was aspirated 11 times to obtain statistically-valid estimates of precision. Background correction (deuterium arc or tungsten halide) was applied to all elements throughout the entire study except those with analytical lines greater than 300.0 nm (Ag, Al, Ba, Cr, Cu).

TABLE 1. CONCENTRATIONS AND WAVELENGTHS USED FOR THE CALIBRATION STUDY

Element	Wavelength (nm)	Analytical Method	Concentrations (mg/L)					Remarks
			Concentration 1	Concentration 2	Concentration 3	Concentration 4	Concentration 5	
Al	309.3	Flame	100	40	20	10	5.0	diluted 1/2 with 2000 ppm K for AA analysis
	308.2	ICP						
Sb	206.8	Flame	50	20	10	5	2.5	
	217.5	ICP						
As	193.7	Graphite furnace	0.5	0.25	0.10	0.05	0.025	contained 1% nickel nitrate
	193.7	ICP						
Ba	553.6	Flame	100	40	20	10	5.0	diluted 1/2 with 2000 ppm K for AA analysis
	493.4	ICP						
Be	234.9	Flame	5	2	1	0.5	0.25	
	313.0	ICP						
Cd	228.8	Flame	5	2	1	0.5	0.25	
	228.8 (X2)*	ICP						second order for ICP
Cr	357.9	Flame	10	4	2	1	0.5	
	267.7	ICP						
Cu	324.8	Flame	10	4	2	1	0.5	
	324.8	ICP						
Fe	248.3	Flame	10	4	2	1	0.5	
	259.9	ICP						
Pb	283.0	Flame	50	20	10	5	2.5	
	220.3	ICP						
Mn	279.5	Flame	5	2	1	0.5	0.25	
	257.6	ICP						
Hg	259.6	Cold-vapor ICP not done	0.01	0.005	0.0025	0.00125	0.0005	

TABLE 1. CONTINUED

Element	Wavelength (nm)	Analytical Method	Concentrations (mg/L) Concentration 1	Concentration 2	Concentration 3	Concentration 4	Concentration 5	Remarks
Ni	232.0	Flame	10	4	2	1	0.5	
	231.6 (X2)*	ICP						second order for ICP
Se	196.0	Graphite furnace	0.5	0.25	0.10	0.05	0.025	contained 1% nickel nitrate
	196.0	ICP						
Ag	328.1	Flame	5	2	1	0.5	0.25	
	328.1	ICP						
Tl	276.8	Flame	50	20	10	5	2.5	
	377.5	ICP						
Zn	213.9	Flame	1	0.40	0.20	0.10	0.05	
	213.9	ICP						

*Second order.

ICP analyses were performed in a similar manner. Each standard was aspirated 11 times. Following analysis of each standard, a distilled, deionized rinse was aspirated. A peristaltic pump was used for sample delivery to minimize changes in aspiration rate due to viscosity differences. After analysis of 15 samples, a check standard was analyzed and the instrument recalibrated if the standard was not within ±5% of the known value. Background correction (spectrum shifter) was applied to all analyses. A Jarrell-Ash Model 970 (multi-channel direct reader) was used for all analyses. An inter-element interference check sample was analyzed after every 15 samples.

Graphite furnace analyses for As and Se were performed using either a Perkin-Elmer Model 603AA or a Model 4000AA both with Model HGA-2100 graphite furnace and Model AS-1 auto sampler. All standards and samples analyzed by graphite furnace contained 1% nickel nitrate. Background correction was used for all analyses. Peak heights, not areas, were employed for calculation of concentrations. Standard additions were not employed. However one sample in ten was spiked to insure recoveries in the range of 90 to 110%.

Mercury analyses by cold vapor AA were performed using a Fisher Mercury Analyzer. This instrument utilizes a pulsed mercury hollow cathode source and a pulsed iron hollow cathode as a background correction source.

Phase 1 Analyses

Phase 1 analyses were performed on samples of river sediment, fly ash, low-pH sludge, and oil/water waste. The Phase 1 procedures consisted of the following general analytical schemes:

Scheme 1 - EP test with leachate digestion

Scheme 2 - EP test without leachate digestion

Scheme 3 - digestion according to EPA procedures[2] with sample stored at room temperature

Scheme 4 - digestion according to EPA procedures[2] with sample preserved by addition of nitric acid (HNO_3) to $pH < 2$

Analytical results from Schemes 1 and 2 were used to evaluate the requirement to digest leachates resulting from the EP test. Results from Schemes 3 and 4 were used to evaluate sample storage requirements.

Samples of river sediment and fly ash were collected on November 31, 1981 and December 3, 1981, respectively. Samples of low-pH sludge and oil/water waste were obtained on November 11, 1982 and August 18, 1982, respectively. The fly ash sample was essentially a dry solid while the other three samples ranged from 50 to 75% aqueous phase. Samples of river sediment, low pH sludge, and oil/water waste were mixed and split into four aliquots - three aliquots were stored at room temperature with no preservation while the fourth aliquot was preserved with concentrated HNO_3 to $pH <2$. Samples of fly ash were split into three aliquots all of which were stored at room temperature (preservation with HNO_3 is not applicable to the dry fly ash). All EP test extractions were performed according to methods outlined by EPA[1]. The EP toxicity test procedure is contained in Appendix J of this report. Digestions for Schemes 1, 3, and 4 were performed according to procedures specified by Section 4.1.3 of reference 2. Modified procedures[2] were utilized for the following elements: As and Se - Methods 206.2 and 270.2; Hg - Method 245.1; and Sb - Method 204.2. Analyses were performed by AAS according to methods specified by EPA[2] and Perkin-Elmer[3]. Graphite furnace AAS was used for atomic absorption determination of As and Se and cold-vapor AAS was used for determination of Hg. All other elements were determined by flame methods. Analyses were also performed by ICP according to methods specified by EPA[4] and Jarrell-Ash[5]. Extractions and analyses were performed in triplicate.

Analyte solutions were spiked by addition of mixed standards to the appropriate dilution of the analyte solution (one part spike to one part analyte). These spikes were analyzed after storage at room temperature for two weeks. The solutions used for spiking were prepared from commercially available AA standards (Fisher or Ventron) or from pure metals (Spex) dissolved in high purity acids and diluted. All spiking solution concentrations were verified by checking them against an EPA EMSL reference water and by analysis at an outside laboratory.

Sample aliquots for Scheme 4 were preserved at the time of collection. After digestion of the samples for Schemes 3 and 4, the analyte solutions were analyzed then spiked. These spikes were stored at room temperature for two weeks and analyzed.

For the oil/water waste (Schemes 1 and 2) the oil phase was removed prior to the performance of the EP procedure. The oil phase was then diluted with xylene and analyzed separately by ICP. Confirming analyses by graphite furnace AAS were performed for elements detected by ICP. Analyses for Hg and Tl could not be performed since organometallic standards and/or suitable methods were unavailable.

The sequence of events occurring for each Scheme of Phase 1 of the project are summarized below:

Scheme 1 - Divide sample aliquot into triplicates, perform EP extraction, digest the extract (separate digestions for Ag, Hg, and Sb), analyze, spike, wait two weeks, analyze by AA.

Scheme 2 - Divide sample aliquot into triplicates, perform EP extraction, analyze, spike, wait two weeks, analyze by AA.

Scheme 3 - Divide sample aliquot into triplicates, digest the triplicates (separate digestions for Ag, Hg, and Sb), analyze, spike, wait two weeks, analyze by AA.

Scheme 4 - Exactly as in Scheme 3 only use the acid preserved sample aliquot.

The analyses performed on the river sediment, fly ash, and low pH sludge are shown schematically in Figure 1 (taken from the Scope of Work). The modified extraction for the oil/water waste is presented schematically in Figure 2 (from the Scope of Work).

Phase 2 Analyses

For the Phase 2 analyses of the low-pH sludge, sixteen separate aliquots were removed, subsampled in triplicate, and adjusted to pH levels of 2, 4, 6, and 8 by addition of 1 M sodium hydroxide (NaOH). At times of 0, 2, 8, and 16 hours following pH adjustment, one triplicate subsample from each pH level was extracted using the EP toxicity test procedure and analyzed for 17 trace metals. Details of extraction and analysis were identical to those described in the section entitled "Phase 1 Analyses," for Scheme 2. The experimental design is presented schematically in Figure 3 (from the Scope of Work).

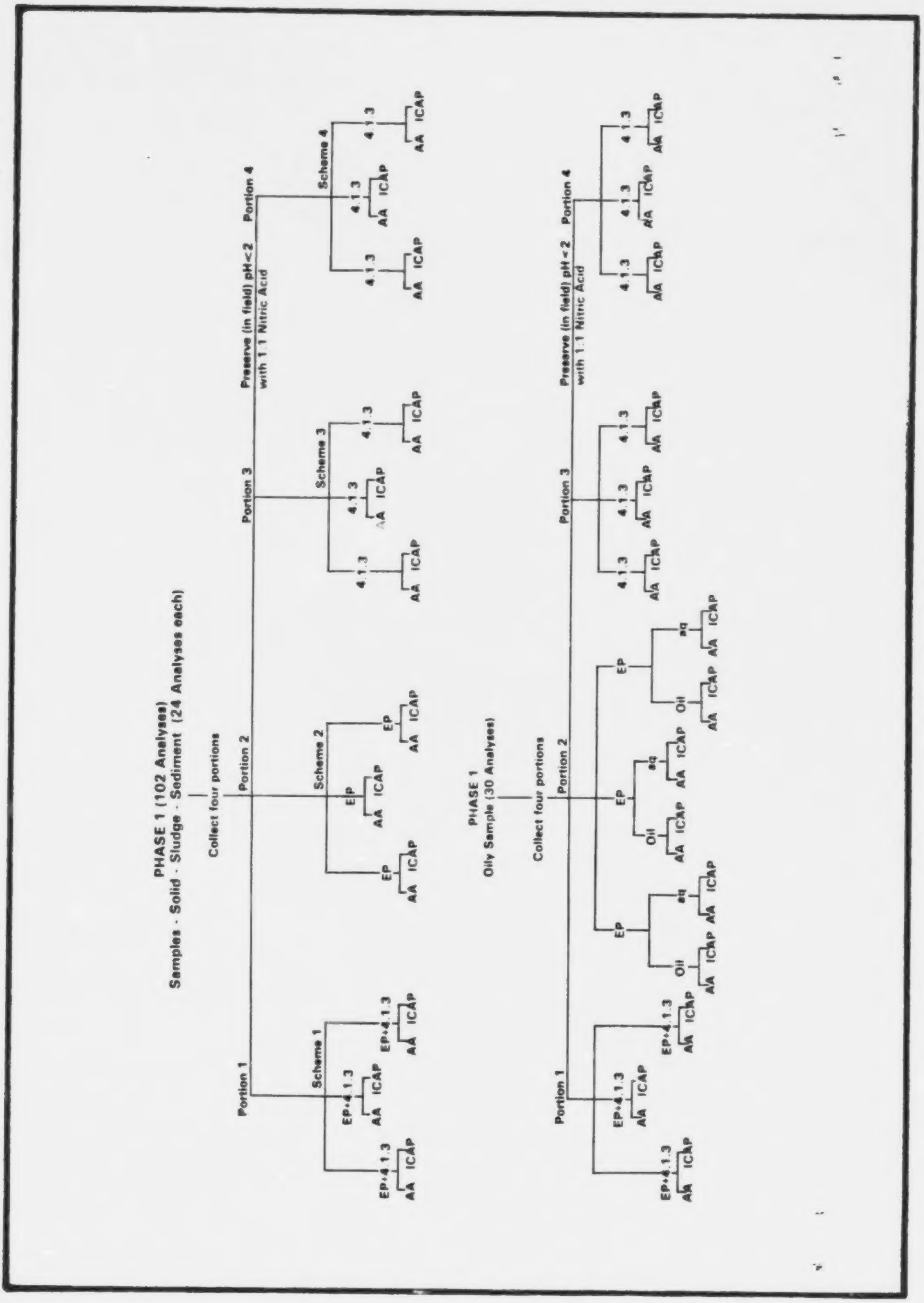
PHASE 1 (102 Analyses)
Samples - Solid - Sludge - Sediment (24 Analyses each)
Collect four portions
Portion 1
Portion 2
Portion 3
Portion 4
Preserve (in field) pH<2 with 1:1 Nitric Acid
Scheme 1
Scheme 2
Scheme 3
Scheme 4
EP+4.1.3
EP
4.1.3
AA
ICAP
PHASE 1
Oily Sample (30 Analyses)
Collect four portions
Portion 1
Portion 2
Portion 3
Portion 4
Preserve (in field) pH<2 with 1:1 Nitric Acid
EP+4.1.3
EP
Oil
aq
4.1.3
AA
ICAP

Figure 1.

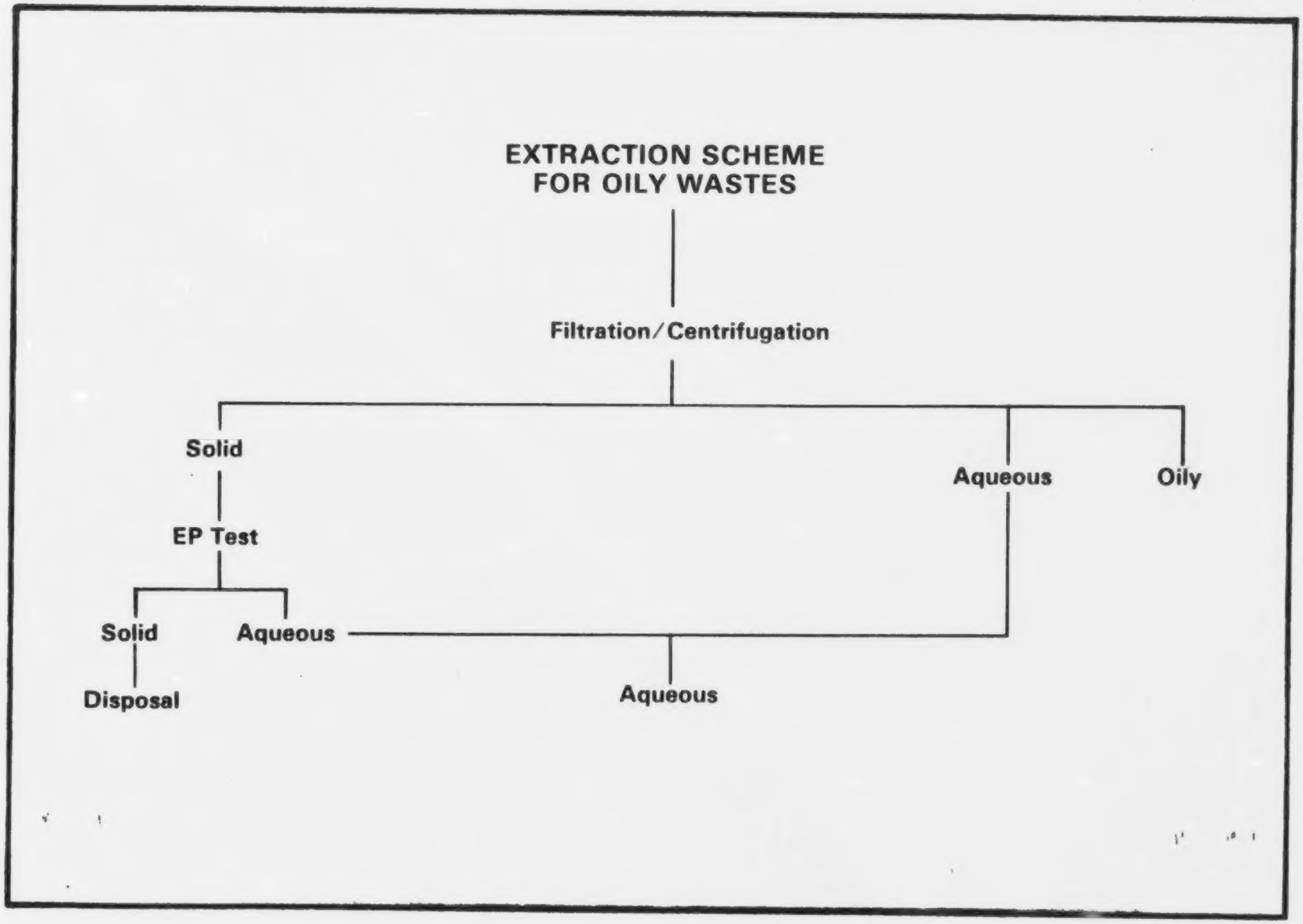
EXTRACTION SCHEME
FOR OILY WASTES
Filtration/Centrifugation
Solid
Aqueous
Oily
EP Test
Solid
Aqueous
Disposal
Aqueous

Figure 2.

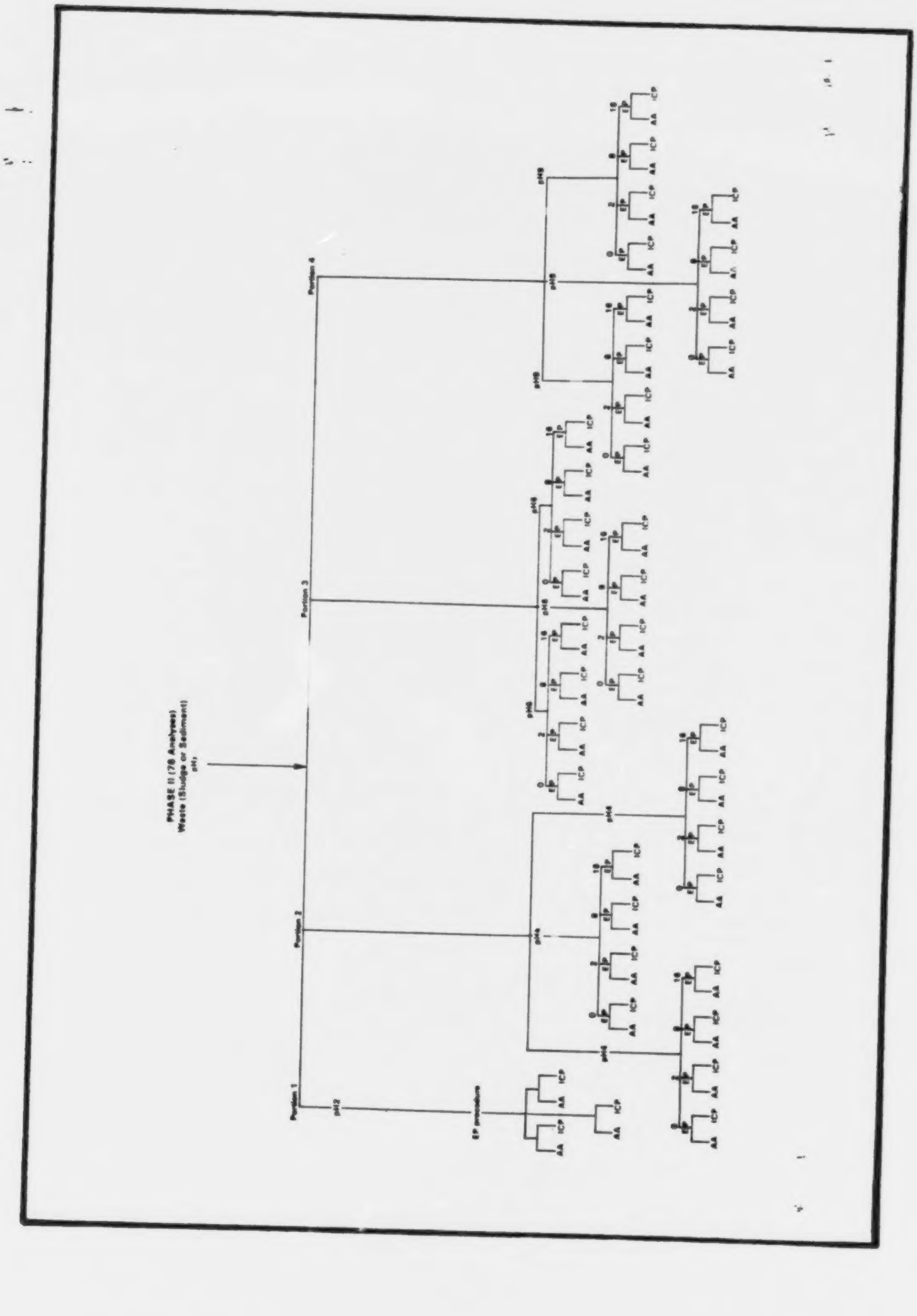
PHASE II (78 Analyses)
Waste (Sludge or Sediment)
pHi
Portion 1
Portion 2
Portion 3
Portion 4
pH2
EP procedure
AA
ICP
EP
0
2
8
10

Figure 3.

Phase 3 Analyses

These standardized reference materials were prepared by drying, grinding, and homogenization of river sediment, fly ash, and EPA Municipal Digested Sludge (the latter material required only homogenization). After determination of total metal concentrations, each sample was divided into ten aliquots and prepared for extraction according to procedures specified for the EP toxicity test. After addition of distilled water, each material was spiked in triplicate at levels approximating 50, 100, and 150% of the total metal concentration using Fisher certified reference standards. An unspiked sample was subjected to the EP test as a control and to allow correction for the background metal concentration in the leachate. During addition of the standard spiking solutions, pH was either allowed to change or was maintained at its initial level by concurrent titration with 0.25 N sodium carbonate (Na_2CO_3). After spike addition, samples were extracted and analyzed using methods identical to those presented in Phase 1, Scheme 2. Phase 3 experiments are depicted schematically in Figure 4 (from the Scope of Work).

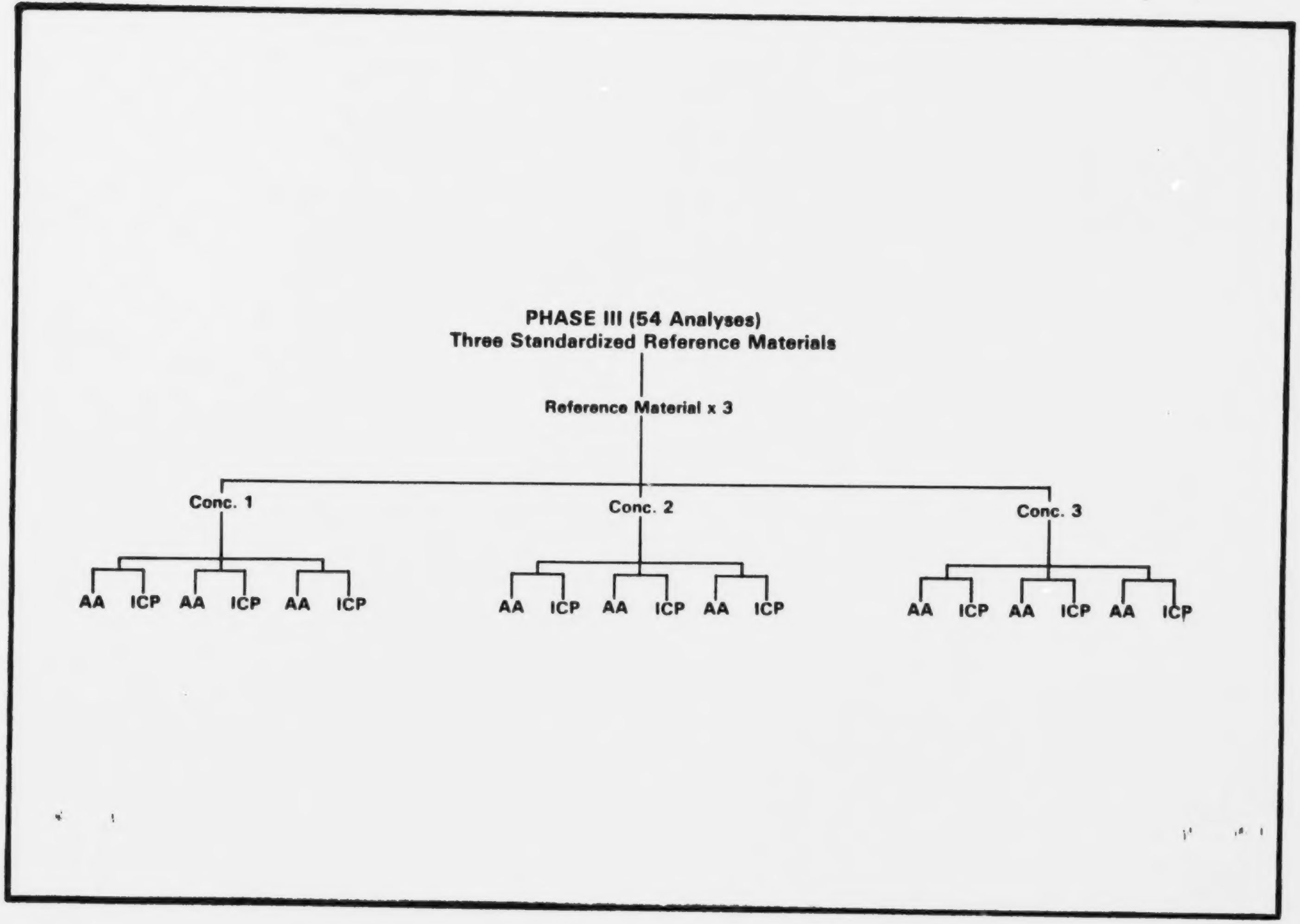
PHASE III (54 Analyses)
Three Standardized Reference Materials
Reference Material x 3
Conc. 1
Conc. 2
Conc. 3
AA
ICP
AA
ICP
AA
ICP
AA
ICP
AA
ICP
AA
ICP
AA
ICP
AA
ICP
AA
ICP

Figure 4.

SECTION 4

RESULTS AND DISCUSSION

CALIBRATION STUDY

Results from the AAS calibration study for 17 elements are summarized in Table 2. For each element, the slope, intercept, and correlation coefficient for the calibration curve are presented. Actual calibration curves are presented in Appendix A. For As, Be, Cd, Hg, and Se, the high standard was in the non-linear range. For these elements, the high standard was omitted from the regression analyses summarized in Table 2.

For all elements except Se, the slopes of the calibration curves for standards prepared in 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid were within ±5% of the slopes of the reference standards prepared in 0.5% nitric acid. No further statistical analyses were performed for these elements since the generally-recommended criteria for check standards requires agreement of ±5% with working standards. In the case of Se, the slope of the calibration curve in the 0.6% acetic acid was 14% greater than the slope of the calibration curve in 0.5% nitric acid. The Se data were analyzed using a t-test to determine if the observed difference in slopes was statistically significant. For comparison of the 0.6% acetic acid with the 0.5% nitric acid reference standard, a calculated t-value of 11.33 (99% confidence level) was obtained versus a tabular value of 2.8, thus indicating a significant difference. For comparison of the 0.6% acetic acid/0.5% nitric acid with the 0.5% nitric acid reference standard, a t-value of 1.7 was obtained, thus indicating a difference that is not significant.

Results from the ICP calibration study for 16 elements are summarized in Table 3. The results are portrayed graphically

TABLE 2. RESULTS OF AAS CALIBRATION STUDY

Element	Regression Equations and Correlation Coefficients for Calibration Curves[a]		
	0.6% acetic acid	0.5% nitric acid	0.6% acetic acid/0.5% nitric acid
Sb	$y = 0.0086x + 0.006$ $r = 0.9997$	$y = 0.0086x + 0.006$ $r = 0.9997$	$y = 0.0087x + 0.005$ $r = 0.9997$
Al	$y = 0.0032x + 0.004$ $r = 0.9998$	$y = 0.0032x + 0.005$ $r = 0.9996$	$y = 0.0032x + 0.005$ $r = 0.9996$
As	$y = 50.75x + 1.56$ $r = 0.9994$	$y = 51.96x + 1.36$ $r = 0.9998$	$y = 51.18x + 1.46$ $r = 0.9992$
Ba	$y = 0.0033x + 0.003$ $r = 0.9999$	$y = 0.0033x + 0.003$ $r = 0.9999$	$y = 0.0033x + 0.004$ $r = 0.9999$
Be	$y = 0.0471x - 0.021$ $r = 0.9940$	$y = 0.0461x - 0.021$ $r = 0.9940$	$y = 0.0461x - 0.022$ $r = 0.9936$
Cd	$y = 0.1500x + 0.006$ $r = 0.9996$	$y = 0.1490x + 0.006$ $r = 0.9997$	$y = 0.1510x + 0.006$ $r = 0.9997$
Cr	$y = 0.0226x + 0.004$ $r = 0.9999$	$y = 0.0225x + 0.002$ $r = 0.9999$	$y = 0.0229x + 0.004$ $r = 0.9999$
Cu	$y = 0.0508x + 0.007$ $r = 0.9997$	$y = 0.0501x + 0.006$ $r = 0.9997$	$y = 0.0509x + 0.007$ $r = 0.9997$
Fe	$y = 0.0269x + 0.004$ $r = 0.9996$	$y = 0.0263x + 0.005$ $r = 0.9996$	$y = 0.0268x + 0.005$ $r = 0.9996$
Pb	$y = 0.0074x + 0.005$ $r = 0.9995$	$y = 0.0073x + 0.006$ $r = 0.9995$	$y = 0.0074x + 0.006$ $r = 0.9995$
Mn	$y = 0.0737x + 0.002$ $r = 0.9999$	$y = 0.0737x + 0.001$ $r = 0.9998$	$y = 0.0751x + 0.001$ $r = 0.9999$
Hg	$y = 0.5349x + 0.008$ $r = 0.9998$	$y = 0.5371x + 0.007$ $r = 0.9997$	$y = 0.5361x + 0.008$ $r = 0.9996$
Ni	$y = 0.0266x + 0.006$ $r = 0.9992$	$y = 0.0261x + 0.005$ $r = 0.9993$	$y = 0.0266x + 0.006$ $r = 0.9994$
Se	$y = 28.28x + 1.13$ $r = 0.9956$	$y = 24.79x + 1.08$ $r = 0.9967$	$y = 25.00x + 0.89$ $r = 0.9981$
Ag	$y = 0.0743x + 0.001$ $r = 0.9999$	$y = 0.0727x + 0.001$ $r = 0.9999$	$y = 0.0738x + 0.001$ $r = 0.9999$
Tl	$y = 0.0101x + 0.010$ $r = 0.9999$	$y = 0.0099x + 0.009$ $r = 0.9999$	$y = 0.0101x + 0.009$ $r = 0.9999$
Zn	$y = 0.2610x + 0.002$ $r = 0.9998$	$y = 0.2616x + 0.002$ $r = 0.9998$	$y = 0.2668x + 0.002$ $r = 0.9998$

[a] Calibration curves were prepared with absorbance or peak height on the y-axis and concentration on the x-axis.

TABLE 3. RESULTS OF ICP CALIBRATION STUDY

Element	Regression Equations and Correlation Coefficients for Calibration Curves[a]		
	0.6% acetic acid	0.5% nitric acid	0.6% acetic acid/0.5% nitric acid
Sb	y = 1.023x + 0.305 r = 0.9999	y = 1.000x + 0.258 r = 0.9999	y = 1.028x + 0.294 r = 0.9999
Al	y = 1.042x + 0.45 r = 0.9999	y = 1.000x + 0.40 r = 0.9999	y = 1.024x + 0.51 r = 0.9999
As	y = 1.041x + 0.007 r = 0.9999	y = 1.000x + 0.010 r = 0.9999	y = 1.047x - 0.064 r = 0.9999
Ba	y = 1.038x - 0.009 r = 0.9999	y = 1.000x - 0.014 r = 0.9999	y = 1.044x - 0.010 r = 0.9999
Be	y = 1.049x - 0.002 r = 0.9999	y = 1.000x - 0.004 r = 0.9999	y = 1.032x - 0.001 r = 0.9999
Cd	y = 0.990x + 0.054 r = 0.9999	y = 1.000x + 0.016 r = 0.9999	y = 1.017x + 0.022 r = 0.9999
Cr	y = 1.030x + 0.30 r = 0.9999	y = 1.000x + 0.023 r = 0.9999	y = 1.028x + 0.034 r = 0.9999
Cu	y = 1.050x + 0.012 r = 0.9999	y = 1.000x - 0.013 r = 0.9999	y = 1.040x - 0.009 r = 0.9999
Fe	y = 1.041x + 0.059 r = 0.9999	y = 1.000x + 0.044 r = 0.9999	y = 1.039x + 0.053 r = 0.9999
Pb	y = 1.031x + 0.208 r = 0.9999	y = 1.000x + 0.147 r = 0.9999	y = 1.023x + 0.148 r = 0.9999
Mn	y = 1.035x + 0.024 r = 0.9999	y = 1.000x + 0.012 r = 0.9999	y = 1.029x + 0.031 r = 0.9999
Ni	y = 0.980x + 0.061 r = 0.9999	y = 1.000x + 0.044 r = 0.9999	y = 1.030x + 0.053 r = 0.9999
Se	y = 1.088x + 0.016 r = 0.9999	y = 1.000x + 0.022 r = 0.9999	y = 1.097x + 0.028 r = 0.9999
Ag	y = 0.985x + 1.1 r = 0.9999	y = 1.000x + 1.0 r = 0.9999	y = 0.987x - 0.30 r = 0.9999
Tl	y = 1.041x - 0.094 r = 0.9999	y = 1.000x + 0.036 r = 0.9999	y = 1.031x - 0.065 r = 0.9999
Zn	y = 1.017x - 0.029 r = 0.9999	y = 1.000x - 0.023 r = 0.9999	y = 1.024x - 0.022 r = 0.9999

[a] Curves were prepared with measured concentration on the y-axis and theoretical concentration on the x-axis. Slopes for standards in 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid were normalized to slope of standards in 0.5% nitric acid. The intercept shown is the actual intercept.

in Appendix A with measured concentration plotted in the y-axis and the theoretical concentration on the x-axis. The slopes summarized in Table 3 were calculated using a linear least squares method and standards prepared in 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid were normalized to the 0.5% nitric acid reference.

For all elements except Se, the slopes of the calibration curves for standards prepared in 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid were within ± 5% of those in the 0.5% nitric acid reference standard. For Se, the slopes in the 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid were higher than the 0.5% nitric acid standard by 8.8 and 9.7%, respectively. These slopes were statistically significant at the 99% confidence level (calculated t-values were 10.8 and 10.2 for the 0.6% acetic acid and 0.6% acetic acid/0.5% nitric acid, respectively, versus a tabulated value of 2.8).

The results from the calibration study indicate that except for selenium, there is no matrix effect from the 0.6% acetic acid or from the 0.6% acetic acid/0.5% nitric acid for either AAS or ICP analytical procedures. Matrix interferences, both enhancements and suppressions, in the graphite furnace analysis of Se have been known since the early developmental stages of the technique. The oxidation state of the Se and the presence of chemical or physical agents which alter or stabilize a particular form of the Se can dramatically alter the Se signal. In particular, the presence of excess metal ions (Ni, Cu) which stabilize the Se as a selenide and the presence of mild reducing agents (sugars, ascorbic acid) also enhance the Se atomization signal[6]. These reducing agents have been postulated to facilitate the reduction of the Se to selenides thereby reducing losses as the volatile SeO_2. Since the nitric/acetic acid mix showed no significant enhancement, it is possible that the oxidizing nitric acid removes the acetic acid or minimizes its effect on the Se.

The presence of acetic acid or the acetic/nitric mixture also enhanced the ICP signal for Se over that in 0.5% nitric alone. While no documentation of this enhancement exists in the scientific literature, the analytical line used in this study is an atom line and it is possible that the acetic acid acted as an ionization suppressor or that a spectral interference was not completely eliminated by the spectral shifter background correction.

The possibility of Se contamination from the acetic acid was considered as a cause for the observed enhancement, but matrix blanks contained negligible amounts of Se (<0.05 ppm). The reported interference by the 1961.25A° line for Fe on the 1960.9A° line for Se has also been considered. Using spectrum shifter for background correction, a negative interference (not an enhancement) of Fe on Se was observed (-0.012 ppm Se/ppm Fe), presumably caused by the spectrum shift onto the Fe line resulting in a negative correction to the Se peak. This interference would decrease the observed Se concentration in the presence of Fe. However, this phenomenon should also occur in the nitric acid matrix which was not observed and, more importantly, no Fe was present in any of the Se calibration standards.

PHASE 1

River Sediment

Results of Phase 1 analyses of river sediment are summarized in Table 4.* Raw data from which these data were abstracted are contained in Appendix B.

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*EP toxicity test leachate results are normally reported as concentration of the metal in the leachate solution. Total extractable metals after an acid digestion are normally reported as concentration in the solid waste.

(continued on next page)

TABLE 4. SUMMARY OF PHASE 1 ANALYSES OF RIVER SEDIMENT

Element	Method	Concentration[a]			
		Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
		mg/L	mg/L	ug/g	ug/g
Al	ICP	0.155 ± 0.099	0.214 ± 0.116	2,730 ± 216	2,650 ± 190
	AAS	0.121 ± 0.107	<0.250[f]	3,053 ± 101	2,746 ± 222
Sb	ICP	<0.010[f]	<0.050[f]	0.94 ± 0.25	1.1 ± 0.28
	AAS	<0.080[f]	<0.500[f]	<10[f]	<10[f]
As	ICP	<0.016[f]	<0.080[f]	5.1 ± 0.23	4.7 ± 0.44
	AAS	<0.002[f]	<0.010[f]	1.6 ± 0.23	0.98 ± 0.02
Ba	ICP	0.164 ± 0.062	0.277 ± 0.075	14 ± 8.1	20 ± 1.0
	AAS	0.151 ± 0.041	<0.250[f]	20 ± 1.0	20 ± 3.5
Be	ICP	0.0004±0.0003	0.0003±0.0002	0.24 ± 0.01	0.25 ± 0.02
	AAS	<0.002[f]	<0.010[f]	0.18 ± 0.01	0.19 ± 0.04
Cd	ICP	0.006 ± 0.002	0.007 ± 0.002	0.34 ± 0.01	0.35 ± 0.03
	AAS	0.006 ± 0.003	<0.010[f]	0.42 ± 0.05	0.37 ± 0.04
Cr	ICP	0.003 ± 0.0	<0.015[f]	18 ± 0.58	18 ± 1.2
	AAS	<0.03[f]	<0.180[f]	18 ± 1.7	17 ± 1.2
Cu	ICP	0.014 ± 0.010	<0.015[f]	16 ± 0.58	17 ± 1.7
	AAS	<0.02[f]	<0.100[f]	19 ± 0.58	20 ± 1.5
Fe	ICP	0.070 ± 0.031	0.088 ± 0.036	4,260 ± 121	4,210 ± 223
	AAS	0.070 ± 0.044	0.123 ± 0.020	4,900 ± 265	3,833 ± 416
Pb	ICP	0.185 ± 0.196	0.232 ± 0.163	383 ± 15	405 ± 61
	AAS	0.160 ± 0.160	<0.200[f]	360 ± 17	383 ± 60
Mn	ICP	1.94 ± 0.81	2.37 ± 0.28	96 ± 1.9	94 ± 3.1
	AAS	2.04 ± 0.83	2.49 ± 0.28	100 ± 0.0	99 ± 1.5
Hg	ICP	<10	<10	<10	<10
	AAS	<0.01	<0.01	0.076 ± 0.013	0.068 ± 0.001
Ni	ICP	0.020 ± 0.010	0.024 ± 0.004	4.1 ± 0.16	4.2 ± 0.02
	AAS	0.019 ± 0.008	<0.050[f]	4.7 ± 0.18	4.5 ± 0.15
Se	ICP	<0.010[f]	<0.050[f]	<5[f]	<5[f]
	AAS	<0.002[f]	<0.010[f]	<0.10[f]	<0.10[f]
Ag	ICP	<0.001[f]	<0.005[f]	0.22 ± 0.02	0.19 ± 0.03
	AAS	<0.005[f]	<0.025[f]	0.29 ± 0.01	0.25 ± 0.03
Tl	ICP	<0.03[f]	<0.15[f]	<2.0[f]	<2.0[f]
	AAS	<0.04[f]	<0.20[f]	<2.0[f]	<2.0[f]
Zn	ICP	0.730 ± 0.487	0.898 ± 0.196	58 ± 1.2	58 ± 2.3
	AAS	0.626 ± 0.389	0.879 ± 0.188	51 ± 1.0	49 ± 1.2

[a] Mean and standard deviation; unless otherwise noted units are mg/L in the extract for Schemes 1 and 2; ug/g for Scheme 3 and 4. Means and standard deviations are for triplicate analyses.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature for two weeks.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2 and stored at room temperature for two weeks.

[f] Mean was not calculated since one or more analyses were below the limit of detection.

The data were analyzed statistically using the unpaired "t" test to determine whether significant differences existed between Schemes 1 and 2 (which compared digested and undigested leachates) and Schemes 3 and 4 (which compared the effect of preservation methods). These results are summarized in Table 5. For the Scheme 1 and 2 data, concentrations of eight elements (Sb, As, Cr, Cu, Hg, Se, Ag, and Tl) were below the limit of detection of the analytical methods and no analyses could be performed. For the other nine elements, (Al, Ba, Be, Cd, Fe, Pb, Mn, Ni, and Zn) no statistically-significant differences were observed between digested and undigested leachates.

For the Scheme 3 and 4 comparisons, only three elements (Sb, Se, and Tl) were present at concentrations below the limit of detection of the analytical methods. For the other 14 elements, significant differences were observed for As, Fe, and Ag using the AAS data, but the ICP data indicated no significant differences for these elements.

Recoveries of spikes from the analyte solutions determined by AAS are summarized in Table 6. Raw data from which these data are abstracted are contained in Appendix B. Recoveries were within the range of 80-120% except for As, Hg, Ag, and Zn, which had recoveries of 109-141%, 66-99%, 73-108% and 47-85%, respectively.

No significant differences were found between the digested and undigested EP leachates for any elements present above the detection

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These conventions have been followed here. In order to compare a value for Schemes 1 and 2 to a value for Scheme 3 or 4 the Scheme 1 or 2 value should be multiplied by a factor of 20 (100 grams of solid brought to a final volume of 2.0 liters of extract with an assumed density of 1.0 g/ml). Detection limits for Schemes 1 and 2 generally differ by a factor of five reflecting the fact that when an extract was digested (Scheme 1) the digestate was not brought back to the original volume but to a volume five times less than the original volume.

TABLE 5. SUMMARY OF STATISTICAL COMPARISONS FOR PHASE 1 ANALYSES OF RIVER SEDIMENT[a,b]

Element	Scheme 1[c] vs Scheme 2[d]	Scheme 3[e] vs Scheme 4[f]
Al	(NS)	NS
Sb	--	-- (NS)
As	--	S (NS)
Ba	(NS)	NS
Be	NS	NS
Cd	NS	NS
Cr	--	NS
Cu	--	NS
Fe	NS	S (NS)
Pb	NS	NS
Mn	NS	NS
Hg	--	NS
Ni	(NS)	NS (NS)
Se	--	--
Ag	--	S (NS)
Tl	--	--
Zn	NS	NS

[a] Unpaired "t" test; confidence interval = 0.95, one-tailed hypothesis, and two degrees of freedom.

[b] All comparisons performed with atomic absorption data unless otherwise noted. Comparisons performed with ICP data are shown in parentheses.

[c] EP toxicity test with leachate digestion.

[d] EP toxicity test without leachate digestion.

[e] Digestion according to EPA[2]. Sample stored at room temperature.

[f] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

-- Not analyzed; one or more data points are below detection limit.
NS Differences not significant (t calc < t tab).
S Differences significant (t calc > t tab).

TABLE 6. SUMMARY OF SPIKE RECOVERIES FROM PHASE 1 ANALYSES OF RIVER SEDIMENT

Element	Average Recovery (%)			
	Scheme 1	Scheme 2	Scheme 3	Scheme 4
Al	97	96	93	92
Sb	92	93	93	92
As	141	137	109	118
Ba	99	100	104	99
Be	103	97	95	106
Cd	97	99	102	100
Cr	106	108	108	144
Cu	98	100	98	100
Fe	104	103	--	--
Pb	100	103	95	117
Mn	106	96	81	80
Hg	97	99	88	66
Ni	97	96	103	100
Se	113	108	99	108
Ag	98	108	105	73
Tl	108	109	113	113
Zn	85	68	47	55

-- Insufficient sample.

limits for the river sediment sample. Significant differences between Schemes 3 and 4 (unpreserved and preserved sample aliquots, respectively) were found by AAS for As, Fe and Ag. However, none of these differences were statistically significant when analyzed by ICP. For all three elements, concentrations as determined by AAS were higher in the unpreserved sample. Consequently, loss to the container walls or losses due to precipitate formation which may be expected in unpreserved samples are not operative here - these would have resulted in lower concentrations for the unpreserved samples. Any chemical or biological degradation of the unpreserved sample resulting in higher concentrations of the three elements should have been detected by ICP as well as AAS. Likewise, sample inhomogeneities would be detected by both techniques.

The iron content of the sample was high and it is possible that errors associated with multiple dilutions may have contributed to the observed differences. Due to the narrower linear range of AAS compared to ICP, more dilutions are required for AAS. In addition, environmental contamination of the aliquots during digestion and dilution is possible for an element as ubiquitous as iron. Contamination during digestion, however, would have been detected by both AAS and ICP.

The results for arsenic by AAS were 0.98 ug/g for the preserved sample and 1.6 ug/g for the unpreserved. The corresponding values for ICP analysis were 4.7 and 5.1 ug/g respectively. Several phenomena are operative here. First, due to the high iron concentration in the sample, a spectral interference is possible particularly near the limit of detection of the ICP. High background scattering at the 196.0 nm wavelength may also cause difficulty in background correction by AAS.

The Ag analyses by AAS were statistically different for Schemes 3 and 4. However, the calculated "t" value was 2.16 while the tabulated value for 95% confidence is 2.13. Given the closeness of these values and the non-significant difference of the ICP data, it is useful to look at the raw data in Table B-1. The triplicate values for AAS Scheme 3 are 0.30, 0.29 and 0.29 ug/g, while those for Scheme 4 are 0.29, 0.24 and 0.24 ug/g. While these numbers are mathematically different, it is doubtful that a practical difference exists between the averages (0.29 ±.01 and 0.25 ±.03).

Recoveries of spikes for all four schemes analyzed by AAS were between 80-120% except for As, Hg, Ag and Zn. Zinc recoveries were consistently low (47-85%), As recoveries consistently high (109-141%). Silver and Hg recoveries were low only for the nitric acid preserved (Scheme 4) samples. The Zn and As recoveries from the fly ash samples also followed these trends indicating a positive interference for As and a loss of Zn to the residual solids or to the glassware.

Fly Ash

Results of Phase 1 analyses of fly ash are summarized in Table 7.* Raw data from which these data were abstracted are contained in Appendix C.

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*EP toxicity test leachate results are normally reported as concentration of the metal in the leachate solution. Total extractable metals after an acid digestion are normally reported as concentration in the solid waste. These conventions have been followed here. In order to compare a value for Schemes 1 and 2 to a value for Scheme 3 or 4 the Scheme 1 or 2 value should be multiplied by a factor of 20 (100 grams of solid brought to a final volume of 2.0 liters of extract with an assumed density of 1.0 g/ml). Detection limits for Schemes 1 and 2 generally differ by a factor of five reflecting the fact that when an extract was digested (Scheme 1) the digestate was not brought back to the original volume but to a volume five times less than the original volume.

TABLE 7. SUMMARY OF PHASE 1 ANALYSES OF FLY ASH

		Concentration[a]			
Element	Method	Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	5.05 ± 0.17	5.43 ± 0.30	8,253 ± 323	---
	AAS	5.62 ± 0.35	5.56 ± 0.00	8,387 ± 179	---
Sb	ICP	<0.010[f]	<0.050[f]	1.9 ± 0.12	---
	AAS	<0.060[f]	<0.080[f]	<10	---
As	ICP	<0.016[f]	<0.080[f]	42 ± 2.1	---
	AAS	0.007 ± 0.001	<0.010[f]	34 ± 3.8	---
Ba	ICP	0.011 ± 0.008	0.165 ± 0.026	132 ± 3.1	---
	AAS	<0.040[f]	<0.250[f]	118 ± 16	---
Be	ICP	0.006 ± 0.001	0.006 ± 0.001	2.46 ± 0.12	---
	AAS	0.005 ± 0.0	<0.025[f]	2.2 ± 0.38	---
Cd	ICP	0.006 ± 0.001	0.007 ± 0.001	<0.05	---
	AAS	0.006 ± 0.001	<0.010[f]	0.25 ± 0.00	---
Cr	ICP	0.021 ± 0.001	0.012 ± 0.001	39 ± 2.1	---
	AAS	<0.030[f]	<0.180[f]	39 ± 5.5	---
Cu	ICP	0.469 ± 0.010	0.454 ± 0.006	27 ± 1.5	---
	AAS	0.465 ± 0.010	0.442 ± 0.010	31 ± 3.0	---
Fe	ICP	0.192 ± 0.085	0.212 ± 0.097	2,630 ± 477	---
	AAS	0.209 ± 0.085	0.255 ± 0.108	3,166 ± 462	---
Pb	ICP	<0.010[f]	<0.010[f]	14 ± 1.0	---
	AAS	<0.040[f]	<0.200[f]	13 ± 1.2	---
Mn	ICP	0.057 ± 0.002	0.057 ± 0.005	19 ± 1.5	---
	AAS	0.055 ± 0.003	0.059 ± 0.007	18 ± 1.5	---
Hg	ICP	---	---	---	---
	AAS	<0.01 ug/l	<0.01 ug/l	0.014 ± 0.001	---
Ni	ICP	0.045 ± 0.001	0.041 ± 0.004	11 ± 1.0	---
	AAS	0.045 ± 0.003	0.062 ± 0.009	13 ± 1.5	---
Se	ICP	<0.010[f]	<0.050[f]	<10	---
	AAS	0.022 ± 0.001	0.016 ± 0.002	5.3 ± 0.87	---
Ag	ICP	<0.001[f]	<0.005[f]	<0.50 [f]	---
	AAS	<0.005[f]	<0.025[f]	<0.50 [f]	---
Tl	ICP	<0.030[f]	<0.150[f]	<2.0	---
	AAS	<0.040[f]	<0.200[f]	<2.0	---
Zn	ICP	0.164 ± 0.012	0.162 ± 0.001	22 ± 1.5	---
	AAS	0.150 ± 0.013	0.143 ± 0.001	24 ± 0.58	

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[a] Mean and standard deviation; unless otherwise noted units are mg/L in the extract for Schemes 1 and 2; ug/g for Scheme 3 and 4. Means and standard deviations are for triplicate analyses.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature for two weeks.

[e] No Scheme 4 analyses were performed on fly ash since preservation with HNO_3 is not applicable to this material.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

--- Not analyzed

The data were analyzed statistically to determine whether significant differences existed between Schemes 1 and 2 (which compared digested and undigested leachates). Analyses for Scheme 4 were not performed for fly ash since preservation with nitric acid is not applicable to this material. Results of the statistical comparisons are summarized in Table 8. For the Scheme 1 and 2 data, concentrations of eight elements (Sb, As, Be, Cr, Pb, Hg, Ag, and Tl) were below the limit of detection of the analytical (AAS) methods and no statistical comparisons could be performed. For four elements (Al, Fe, Mn, and Zn), no significant differences were detected. Significant differences were detected for Ba, Cd, and Cr (using ICP data) and for Cd, Ni, Cu, and Se using the AAS data. Differences for Cu and Ni were not significant using the ICP data.

Recoveries of spikes from the analyte solutions are summarized in Table 9. Recoveries of 11 elements (Al, Sb, Be, Cd, Cu, Fe, Mn, Hg, Ni, Ag, and Tl) were in the range of 80-120%. Recoveries for the remaining six elements were as follows: As, 116-143%; Ba, 0-108%; Cr, 108-128%; Pb, 70-101%; Se, 30-95%; and Zn, 70-88%.

Significant differences for fly ash EP leachates were found for Cu, Ni and Se by AAS and for Ba, Cd and Cr by ICP. Differences for Cu and Ni were not significant by ICP and Se was below the ICP detection limit. Cu and Ni by AA are affected by Fe and other transition metals (7). The difference in Ba concentrations between the digested and undigested leachates is over an order of magnitude (the undigested value higher). Either a spectral interference is eliminated by the digestion or neutral or reduced sulfur species (the fly ash was high in sulfur) were oxidized by the digestion and precipitated $BaSO_4$. The AAS data for both Schemes 1 and 2 were below the detection limit and cannot be used

TABLE 8. SUMMARY OF STATISTICAL COMPARISONS OF PHASE 1 ANALYSES FOR FLY ASH[a,b]

Element	Scheme 1 vs[c] Scheme 2[d]	Scheme 3 vs[e] Scheme 4[f]
Al	NS	Not Applicable[g]
Sb	--	
As	--	
Ba	(S)	
Be	--	
Cd	S (S)	
Cr	(S)	
Cu	S (NS)	
Fe	NS	
Pb	--	
Mn	NS	
Hg	--	
Ni	S (NS)	
Se	S	
Ag	--	
Tl	--	
Zn	NS	

[a] Unpaired "t" test; confidence interval = 0.95, one-tailed hypothesis, and two degrees of freedom.

[b] All comparisons performed with atomic absorption data unless otherwise noted. Comparisons performed with ICP data are shown in parentheses.

[c] EP toxicity test with leachate digestion.

[d] EP toxicity test without leachate digestion.

[e] Digestion according to EPA[2]. Sample stored at room temperature.

[f] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

[g] Preservation with HNO_3 is not applicable to this material.

-- Not analyzed; one or more data points are below detection limit.

NS Differences not significant (t calc < t tab).

S Differences significant (t calc > t tab).

TABLE 9. SUMMARY OF SPIKE RECOVERIES FROM PHASE 1 ANALYSES OF FLY ASH

Element	Average Recovery (%) Scheme 1	 Scheme 2	 Scheme 3
Al	96	97	83
Sb	101	96	99
As	126	116	143
Ba	0	108	95
Be	100	98	97
Cd	98	97	96
Cr	111	108	128
Cu	98	101	96
Fe	102	107	94
Pb	70	101	100
Mn	107	90	117
Hg	87	114	106
Ni	100	96	104
Se	30	67	95
Ag	86	104	103
Tl	116	112	118
Zn	87	88	70

to support either possibility. However, Ba was not recovered from the digested leachate implying that the precipitation of $BaSO_4$ is the likely mechanism.

The Cd difference is solely a mathematical consequence of the high precision at the low concentrations and the difference between 0.006 mg/L and 0.007 mg/L is of no practical importance. The difference in Cr values by ICP (0.021 mg/L digested, 0.012 mg/L undigested) is large but is near the ICP detection limit and is at a level where contamination of the sample during digestion is entirely possible and therefore of no practical importance.

The undigested (Scheme 2) leachate yielded a Cu value by AAS which is significantly lower than the digested value and lower than the ICP value for either the digested or undigested leachates. The Ni value for the undigested leachate was significantly higher than either the ICP value or the digested leachate AAS concentration. These concentrations are not near the detection limits of either technique and show that the AAS technique is subject to both positive (Ni) and negative (Cu) interferences without digestion of the leachate. The differences in Se values by AAS are statistically significant but are near the detection limit of the graphite furnace technique. Although the precision of the measurements is quite high (consequently the significant difference), the possibility of contamination at these levels must not be forgotten when evaluating the practical significance of the difference.

As with the river sediment samples, Zn recoveries were consistently low, As recoveries consistently high. In addition, Cr recoveries were somewhat high particularly for the completely digested (Scheme 3) sample supporting the possibility of contamination during digestion.

Recoveries of Ba, Pb, Se and Ag were low for the digested EP leachate (Scheme 1). The fly ash sample was generated from fuel containing significant levels of sulfur and the possibility of sulfate formation during digestion with subsequent precipitation of insoluble sulfates (Ba, Pb, Ag) is highly likely, the low Se recovery cannot be explained by sulfate precipitation but may be due to the formation of nickel sulfides rather than nickel selenides in the graphite furnace with subsequent loss of SeO_2.

Low pH Sludge

Results of Phase 1 analyses of low-pH sludge are summarized in Table 10.* Raw data from which these data were abstracted are contained in Appendix D.

The data were analyzed statistically to determine whether significant differences existed between Schemes 1 and 2 (which compared digested and undigested leachates) and Schemes 3 and 4 (which compared the effect of preservation methods). Results of statistical analyses are summarized in Table 11. For the Scheme 1 and 2 data, concentrations of seven elements (Sb, Ba, Be, Hg, Se, Ag, and Tl) were below the limit of detection of the analytical methods and no statistical comparisons could be performed. For

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*EP toxicity test leachate results are normally reported as concentration of the metal in the leachate solution. Total extractable metals after an acid digestion are normally reported as concentration in the solid waste. These conventions have been followed here. In order to compare a value for Schemes 1 and 2 to a value for Scheme 3 or 4 the Scheme 1 or 2 value should be multiplied by a factor of 20 (100 grams of solid brought to a final volume of 2.0 liters of extract with an assumed density of 1.0 g/ml). Detection limits for Schemes 1 and 2 generally differ by a factor of five reflecting the fact that when an extract was digested (Scheme 1) the digestate was not brought back to the original volume but to a volume five times less than the original volume.

TABLE 10. SUMMARY OF PHASE 1 ANALYSES OF LOW-pH SLUDGE

Element	Method	Concentration[a]			
		Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	15±1	15±0	1840±90	1880±110
	AAS	16±1	15±1	1870±180	1960±130
Sb	ICP	<0.053[f]	<0.200[f]	<49[f]	<54[f]
	AAS	<0.053[f]	<0.200[f]	<1.9[f]	<2.1[f]
As	ICP	<2.0[f]	0.470±0.049	<18[f]	<21[f]
	AAS	0.056±0.009	0.092±0.005	1.9±0.1	2.0±0.1
Ba	ICP	0.151±0.018	0.126±0.018	22±1	25±4
	AAS	<0.500[f]	<0.500[f]	26±2	24±3
Be	ICP	<0.003[f]	<0.010[f]	<2.4[f]	<2.7[f]
	AAS	<0.013[f]	<0.010[f]	<0.100[f]	<0.110[f]
Cd	ICP	0.32±0.02	0.28±0.05	2.7±0.1	2.5±0.2
	AAS	0.24±0.01	0.23±0.01	2.7±0.1	2.5±0.1
Cr	ICP	15±1	15±0	1380±0	1340±10
	AAS	16±1	17±1	1360±30	1360±40
Cu	ICP	2.4±0.1	2.6±0.2	37±1	34±1
	AAS	2.3±0.2	2.1±0.2	32±1	32±0
Fe	ICP	1570±60	1590±30	29,200±100	27,700±100
	AAS	1570±60	1580±40	28,900±200	28,500±100
Pb	ICP	5.1±0.4	4.6±0.3	137±3	126±6
	AAS	4.2±0.1	4.1±0.1	117±1	116±1
Mn	ICP	30±1	30±1	381±0	368±1
	AAS	31±2	30±1	380±2	372±3
Hg	ICP	--	--	--	--
	AAS	<0.0001[f]	<0.0001[f]	0.10±0.03	0.10±0
Ni	ICP	77±2	74±2	960±7	924±11
	AAS	81±2	75±1	941±1	931±5
Se	ICP	<50[f]	<11[f]	<275[f]	<312[f]
	AAS	<0.050[f]	<0.050[f]	<0.46[f]	<0.52[f]
Ag	ICP	<0.100[f]	<0.010[f]	<0.92[f]	<0.98[f]
	AAS	<0.010[f]	<0.010[f]	<0.23[f]	<0.24[f]
Ti	ICP	<5.0[f]	<1.0[f]	<46[f]	<49[f]
	AAS	<0.080[f]	<0.200[f]	<1.8[f]	<2.0[f]
Zn	ICP	3670±170	3460±130	40,600±600	39,800±400
	AAS	3950±150	3140±90	39,900±800	39,100±400

[a] Mean and standard deviation; unless otherwise noted units are mg/L in the extract for Schemes 1 and 2; ug/g for Scheme 3 and 4. Means and standard deviations are for triplicate analyses.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature for two weeks.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2 and stored at room temperature for two weeks.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

-- Not analyzed.

TABLE 11. SUMMARY OF STATISTICAL COMPARISONS OF PHASE 1 ANALYSES FOR LOW pH SLUDGE[a,b]

Element	Scheme 1[c] vs Scheme 2[d]	Scheme 3[e] vs Scheme 4[f]
Al	NS	NS
Sb	--	--
As	S	NS
Ba	--	NS
Be	--	--
Cd	S (NS)	S (NS)
Cr	NS	NS
Cu	NS	NS (S)
Fe	NS	S (S)
Pb	NS	NS (S)
Mn	NS	S (S)
Hg	--	NS
Ni	S (NS)	S (S)
Se	--	--
Ag	--	--
Tl	--	--
Zn	S (NS)	NS

[a] Unpaired "t" test; confidence interval = 0.95, one-tailed hypothesis, and two degrees of freedom.

[b] All comparisons performed with atomic absorption data unless otherwise noted. Comparisons performed with ICP data are shown in parentheses.

[c] EP toxicity test with leachate digestion.

[d] EP toxicity test without leachate digestion.

[e] Digestion according to EPA[2]. Sample stored at room temperature.

[f] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

-- Not analyzed; one or more data points are below detection limit.
NS Differences not significant (t calc < t tab).
S Differences significant (t calc > t tab).

6 elements (Al, Cr, Cu, Fe, Pb, and Mn), no significant differences were detected. For four elements (As, Cd, Ni, and Zn), significant differences were detected using AAS results. For Cd, Ni, and Zn, differences were not significant using ICP data. For the Scheme 3 and 4 data, which compared preserved and unpreserved sample aliquots, five elements (Sb, Be, Se, Ag and Tl) were below the limits of detection. By AAS, no significant differences were detected for Al, As, Ba, Cd, Cu, Pb, Mn and Zn. Significant differences were detected for Cd, Fe, Mn and Ni by AAS and for Cu, Fe, Pb, Mn and Ni by ICP.

Recoveries of spikes from the analyte solutions are summarized in Table 12. For 11 elements (Al, Sb, Be, Cd, Cr, Cu, Fe, Mn, Hg, Ni, and Tl), recoveries were in the range of 80-120%. Recoveries for the remaining elements were as follows: As, 56-92%; Ba, 0%; Pb, 41-89%; Se, 78-98%; Ag, 7-24%; and Zn, 86-128%.

AAS data for Schemes 1 and 2 indicate significant differences for As, Cd, Ni, and Zn. Arsenic levels were below ICP detection limits while the ICP data for Cd, Ni, and Zn showed no differences. The As values for the undigested leachate are higher than for the digested sample. Two possible causes are that As is lost during digestion or that there is a spectral interference in the undigested sample. Spike recoveries for the undigested sample averaged 56% and for the digested sample 84% supporting the likelihood of an interference in the undigested sample.

The practical significance of the AAS Cd data is suspect. The high precision allows 0.23 and 0.24 mg/L to be statistically different.

The nickel data for the digested leachate are higher (by both AAS and ICP although not significant by ICP). Since the

TABLE 12. SUMMARY OF SPIKE RECOVERIES FROM PHASE 1 ANALYSES OF LOW-pH SLUDGE

Element	Average Recovery (%) Scheme 1	Scheme 2	Scheme 3	Scheme 4
Al	95	95	97	91
Sb	97	98	96	98
As	84	56	92	89
Ba	0	0	0	0
Be	82	90	93	91
Cd	98	99	98	99
Cr	111	106	113	114
Cu	94	97	98	98
Fe	100	92	95	95
Pb	41	89	52	54
Mn	99	99	101	100
Hg	96	96	84	100
Ni	85	91	88	88
Se	92	78	98	94
Ag	22	24	7	9
Tl	102	104	104	101
Zn	86	128	99	98

difference is small, contamination during digestion or slight inhomogeneities in the sample aliquots are the likely causes. An enhancement of flame AAS analyses for Ni by large excesses of Fe is well known (7).

The leachable Zn content of this sample is extremely high and the values for the digested leachate are higher by both AAS and ICP than the undigested leachate, although (as with Ni) the ICP data are not statistically higher. Contamination during digestion, errors from multiple dilutions, sample inhomogeneity or a negative interference in the undigested leachate are all possible causes.

In the comparison of Schemes 3 and 4, the AAS data is significantly different for Cd but the ICP data is not. For Cu, the ICP data is significantly different while the AAS data is not. An examination of the raw data (Table D-1) again suggests that these results are the mathematical consequence of high precision rather than of a practical difference. The Pb data for ICP Scheme 3 (unpreserved) is higher than for the preserved sample while the AAS data shows no difference. Both the Scheme 3 and Scheme 4 ICP values are higher than the AA results. The most likely explanation of this is that it is a consequence of a spectral interference from the extremely high levels of Fe, Mn or Ni present in this sample. If the difference were due to a true chemical change in the unpreserved sample or a sample inhomogeneity, the AAS results should also reflect the increased concentration.

Three elements (Fe, Mn and Ni) showed statistically significant differences for preserved and unpreserved samples by both AAS and ICP. For all three elements, the concentrations found in the unpreserved sample are higher than for the preserved sample and for all three elements, the concentrations are high. This waste is a sludge from a steel acid "pickling bath" and the high concentrations of these and other transition metals is expected. However, this waste had a high residual sulfuric acid content (a "low-pH sludge") and was, therefore, "acid preserved" to some extent even without the addition of nitric acid. The data strongly suggest that the nitric acid preservation caused changes in the chemical form of some transition metals and that these were not solubilized by the acid digestion of the sludge or that they were lost in digestion.

Recoveries of spikes from this waste were generally high with two exceptions, As and Se recoveries from the undigested EP leachate were low and elements forming insoluble sulfates had universally poor recoveries (Ba, Pb, and Ag).

Oil/Water Waste

Results of Phase 1 analyses of the oil/water waste are shown for the aqueous phase in Table 13* and for the oil phase in Table 14. Raw data from which data were abstracted are contained in Appendix E.

The data from the aqueous phase analyses were analyzed statistically to determine whether significant differences

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*EP toxicity test leachate results are normally reported as concentration of the metal in the leachate solution. Total extractable metals after an acid digestion are normally reported as concentration in the solid waste. These conventions have been followed here. In order to compare a value for Schemes 1 and 2 to a value for Scheme 3 or 4 the Scheme 1 or 2 value should be multiplied by a factor of 20 (100 grams of solid brought to a final volume of 2.0 liters of extract with an assumed density of 1.0 g/ml). Detection limits for Schemes 1 and 2 generally differ by a factor of five reflecting the fact that when an extract was digested (Scheme 1) the digestate was not brought back to the original volume but to a volume five times less than the original volume.

TABLE 13. SUMMARY OF PHASE 1 ANALYSES OF OIL/WATER WASTE, AQUEOUS PHASE

Element	Method	Concentration[a]			
		Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	0.145 ± 0.057	0.103 ± 0.058	7980 ± 720	7820 ± 1100
	AAS	0.159 ± 0.064	<0.500[f]	7640 ± 730	7760 ± 1000
Sb	ICP	<0.040[f]	<0.200[f]	<16[f]..	<17[f]
	AAS	<0.100[f]	<0.500[f]	<41[f]	<44 [f]
As	ICP	<0.040[f]	<0.200[f]	<18[f]..	<17[f]
	AAS	0.003 ± 0.001	<0.005[f]	6.8 ± 0.9	7.2 ± 1.7
Ba	ICP	0.017 ± 0.005	0.022 ± 0.015	87 ± 3	87 ± 17
	AAS	<0.100[f]	<0.500[f]	78 ± 8	72 ± 16
Be	ICP	<0.002[f]	<0.010[f]	<0.82[f]	<0.87[f]
	AAS	<0.001[f]	<0.005[f]	0.36 ± 0.04	0.38 ± 0.1
Cd	ICP	<0.002[f]	<0.010[f]	<0.82[f]	<0.87[f]
	AAS	<0.002[f]	<0.010[f]	0.74 ± 0.03	0.95 ± 0.17
Cr	ICP	<0.005[f]	<0.025[f]	24 ± 3	23 ± 1
	AAS	<0.010[f]	<0.050[f]	28 ± 4	28 ± 2
Cu	ICP	<0.010	<0.050	29 ± 11	29 ± 6
	AAS	<0.004[f]	<0.020[f]	20 ± 5	24 ± 6
Fe	ICP	0.450 ± 0.047	0.787 ± 0.370	13,700 ± 1490	14,400 ± 960
	AAS	0.458 ± 0.048	0.700 ± 0.300	14,200 ± 1490	14,830 ± 1150
Pb	ICP	<0.040[f]	<0.200[f]	106 ± 21	105 ± 21
	AAS	<0.020[f]	<0.100[f]	95 ± 18	95 ± 26
Mn	ICP	0.080 ± 0.007	0.084 ± 0.035	90 ± 16	80 ± 6
	AAS	0.085 ± 0.006	0.084 ± 0.046	90 ± 18	80 ± 6
Hg	ICP	<10	<10	<10	<10
	AAS	<0.001[f]	<0.001[f]	0.010 ± 0.004	0.009 ± 0.001
Ni	ICP	0.014 ± 0.003	<0.050[f]	12 ± 0	11 + 2
	AAS	<0.010[f]	<0.050[f]	11 ± 0	11 + 1
Se	ICP	<0.020[f]	<0.100[f]	<180[f]	<170 [f]
	AAS	<0.005[f]	<0.005[f]	<0.090[f]	<0.084 [f]
Ag	ICP	<0.002[f]	<0.010[f]	<0.085[f]	<0.086[f]
	AAS	<0.002[f]	<0.010[f]	<0.085[f]	<0.086[f]
Tl	ICP	<0.100[f]	<0.500[f]	<43	<43[f]
	AAS	<0.020[f]	<0.100[f]	<0.85[f]	<0.86[f]
Zn	ICP	0.087 ± 0.014	0.062 ± 0.043	105 ± 9	109 ± 9
	AAS	0.082 ± 0.011	0.043 ± 0.048	96 ± 6	102 ± 8

[a] Mean and standard deviation; unless otherwise noted units are mg/L in the extract for Schemes 1 and 2; ug/g for Scheme 3 and 4. Means and standard deviations are for triplicate analyses.

[b] EP toxicity test with leachate digestion; aqueous phase only.

[c] EP toxicity test without leachate digestion; aqueous phase only.

[d] Digestion according to EPA[2]. Sample stored at room temperature for two weeks.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2 and stored at room temperature for two weeks.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

-- Not analyzed.

TABLE 14. SUMMARY OF PHASE 1 ANALYSIS OF OIL/WATER WASTE - OIL PHASE

Element	Concentration[a] Scheme 1[b]	 Scheme 2[c]	% of Total Metal Scheme 1	 Scheme 2
Al	0.071±0.006	0.13±0.02	32	57
Sb	<0.015	<0.015	--	--
As	<0.015	<0.015	--	--
Ba	0.0016±0.0004	0.0030±0.0003	9	12
Be	<0.001	<0.001	--	--
Cd	<0.001	<0.001	--	--
Cr	<0.0025	<0.0026	--	--
Cu	0.0021±0.0010	0.0069±0.0004	--	--
Fe	0.16±0.07	0.29±0.03	26	29
Pb	0.021±0.003	0.048±0.010	--	--
Mn	0.016±0.002	0.026±0.003	17	24
Hg	--	--	--	
Ni	0.011±0.001	0.017±0.001	44	--
Se	<0.10	<0.10	--	--
Ag	<0.001	<0.001	--	--
Tl	--	--	--	--
Zn	<0.001	<0.001	--	--

[a] Mean and standard deviation; unless otherwise noted, units are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion; oil phase only.

[c] EP toxicity test without leachate digestion; oil phase only.

-- Not analyzed

existed between Schemes 1 and 2 (which compared digested and undigested leachates) and Schemes 3 and 4 (which compared the effect of preservation method). Results of statistical analysis are shown in Table 15. For data from Schemes 1 and 2, only two elements (Fe and Mn) were present at concentrations above the limit of detection and no significant differences were evident. For data from Schemes 3 and 4, all elements except Sb, As, Se, Ag, and Tl were present at concentrations above the limit of detection and no significant differences were detected.

Oil phase analyses (Table 14) indicated measurable amounts of Al, Ba, Cu, Fe, Pb, Mn, and Ni in the leachates. For purposes of comparison, concentrations were calculated on the same basis as the previous phase leachate. The fractions of the total metal contained in the oil phase were as follows: Al, 32-57%; Ba, 9-12%; Fe, 26-29%; Mn, 17-24%; and Ni, 44%.

Recoveries of spikes from the analyte solutions are summarized in Table 16. For 15 elements (Sb, As, Ba, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Se, Ag, Tl, and Zn), recoveries were in the range of 80-120%. Recoveries of the remaining elements were as follows: Al, 96-124%; and Hg, 15-104%.

No significant differences were found for this waste by either ICP or AAS. The precision of the analysis for this waste was much poorer than for any other waste probably due to incomplete separation of the two phases.

Recoveries of Hg for the digestion of both preserved and unpreserved samples were low indicating a loss during digestion. Recoveries of Al for the preserved and unpreserved samples (Schemes 3 and 4) were high.

TABLE 15. SUMMARY OF STATISTICAL COMPARISONS OF PHASE 1 ANALYSES FOR OIL/WATER WASTE[a,b]

Element	Scheme 1[c] vs Scheme 2[d]	Scheme 3[e] vs Scheme 4[f]
Al	--	NS
Sb	--	--
As	--	--
Ba	--	NS
Be	--	NS
Cd	--	NS
Cr	--	NS
Cu	--	NS
Fe	NS	NS
Pb	--	NS
Mn	NS	NS
Hg	--	NS
Ni	--	NS
Se	--	--
Ag	--	--
Tl	--	--
Zn	--	NS

[a] Unpaired "t" test; confidence interval = 0.95, one-tailed hypothesis, and two degrees of freedom.

[b] All comparisons performed with atomic absorption data unless otherwise noted. Comparisons performed with ICP data are shown in parentheses.

[c] EP toxicity test with leachate digestion (aqueous phase only).

[d] EP toxicity test without leachate digestion (aqueous phase only).

[e] Digestion according to EPA[2]. Sample stored at room temperature.

[f] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

-- Not analyzed; one or more data points are below detection limit.
NS Differences not significant (t calc < t tab).
S Differences significant (t calc > t tab).

TABLE 16. SUMMARY OF SPIKE RECOVERIES FROM PHASE 1 ANALYSES OF OIL/WATER WASTE

Element	Average Recovery (%)			
	Scheme 1	Scheme 2	Scheme 3	Scheme 4
Al	96	99	124	119
Sb	103	102	102	106
As	96	101	103	104
Ba	102	102	119	107
Be	100	99	98	96
Cd	102	101	102	102
Cr	106	103	101	104
Cu	101	101	103	106
Fe	108	103	108	107
Pb	100	99	105	108
Mn	103	104	105	105
Hg	103	104	15	19
Ni	104	103	103	103
Se	119	110	101	109
Ag	103	100	109	110
Tl	103	101	109	110
Zn	102	101	99	99

PHASE 2 ANALYSES

Raw data for the Phase 2 analysis of the low pH sludge are contained in Appendix F. Aliquots of the sludge were adjusted to pH 2, 4, 6, and 8 with 1 M NaOH and then extracted at time intervals (post-pH adjustment) of 0, 2, 8 and 16 hours. Of the 17 elements in this study, 10 were present in the pH 2 samples at concentrations above the analytical detection limits (Al, As, Cd, Cr, Cu, Fe, Pb, Mn, Ni and Zn).

None of the metals exhibited a strong leachate concentration dependence on the time interval between pH adjustment and extraction. However, nine of the ten elements present (As being the single exception) exhibited strong concentration dependence on pH during the EP leachate generation. Given the nature of the low pH sludge, these results are not surprising. The waste, from a steel "pickling bath" contains high levels of iron and other transition metals and high concentrations of sulfate. Consequently, any element forming insoluble sulfates or hydroxides would be lost at higher pH. Arsenic, which would be present as oxyanion species, would not form insoluble hydroxides or sulfates and would be lost only by mechanisms such as co-precipitation. In fact since the concentrations of iron, aluminum, zinc, and sulfate are high in this waste, it is surprising that As was not lost via co-precipitation.

It should be noted that the aliquots of sludge which were adjusted to pH 6 or 8 were (at the appropriate time interval) adjusted to pH 5 as would be required by the EP test (1). The extracts were analyzed without digestion, that is, by procedures analogous to Phase 1, Scheme 2.

PHASE 3 ANALYSES

The purpose of Phase 3 was to determine the efficiency of the EP procedure in extracting metal spikes from standard reference materials. The materials chosen were fly ash, river sediment, and EPA Municipal Digested Sludge. The fly ash and river sediment were dried, ground, and homogenized prior to use. The samples of dried municipal sludge were composited to obtain a sufficient quantity of homogenous material for the study.

Aliquots of all three materials were digested and analyzed in triplicate by AAS to provide the total bulk concentrations of the 17 metals in the reference materials. The results obtained by ERCO are presented in Table 17 and were confirmed by an outside laboratory.

Each sample was prepared for the EP extraction according to the EPA-specified methods (1). After addition of distilled water to the dried ash, sediment and sludge, the samples were spiked (in triplicate) at three different concentration levels representing 150%, 100%, and 50% of the total metal content or 20, 10, and 5 times the detection limit for those elements which were not detected in the bulk analyses of the materials. Due to the acid content of the spiking solutions, the pH of the spiked sample/distilled water matrices ranged from 1.39 to 3.28. This pH range is low for EP extractions where the pH of neutral or basic sludges are typically adjusted to pH 5.0 ±0.2 with acetic acid.

After discussions with the EPA Project Officer, the decision was made to spike and analyze a second series of samples. For this series, the sample pH was maintained at its initial level during spike addition by concurrent titration with 0.25 N Na_2CO_3. These samples were then adjusted to pH 5 and extracted according to the EP test procedure (1).

TABLE 17. TOTAL CONCENTRATIONS OF METALS IN REFERENCE MATERIALS

	Concentration (μg/g, dry weight)[a]		
Element	**Municipal Digested Sludge**	**Fly Ash**	**River Sediment**
Ag	51.9 ± 1.5	<0.5	<0.5
Al	4,030 ± 71	14,100 ± 1,960	5,670 ± 568
As	5.0 ± 1.5	34.5 ± 8.6	5.1 ± 4.1
Ba	305 ± 235	190 ± 19	46.5 ± 3.6
Be	0.23 ± 0.10	3.9 ± 0.55	0.52 ± 0.03
Cd	15.0 ± 0.76	<0.5	0.62 ± 0.07
Cr	169 ± 7.4	47.5 ± 6.5	34.6 ± 2.5
Cu	1,040 ± 47	35.3 ± 2.8	30.5 ± 3.3
Fe	16,000 ± 401	4,840 ± 1,200	9,160 ± 600
Mn	183 ± 7.2	27.4 ± 4.8	167 ± 8.7
Ni	155 ± 6.7	18.5 ± 4.1	8.1 ± 0.46
Pb	421 ± 29	6.75 ± 1.25	810 ± 63
Sb	<0.90	<0.20	<0.60
Se	<0.70	3.4 ± 0.4	<0.5
Tl	<0.70	3.07 ± 2.55	0.30 ± 0.12
Zn	1,050 ± 47	29.9 ± 6.0	89.6 ± 7.2

---

[a] Mean and standard deviation based on triplicate analyses

The EP toxicity test is designed to determine the mobility of hazardous constituents in a waste, not to extract the total amount of a constituent. Consequently an unspiked sample was prepared and extracted with each of the spike samples. For the pH adjusted series, the pH of the unspiked samples were 5.0 ±0.2 while for the high acid containing samples, the pH was adjusted to the lowest value encountered within each reference material sample set. In addition, for each spike level, a "blank spike" was prepared by adding the spikes to distilled water. This was utilized to verify the integrity of the sample spikes. At least one extract from each sample matrix (fly ash, river sediment, and sludge) was spiked during analysis, for both pH adjusted and non-adjusted samples, to determine analytical recoveries and to verify concentrations determined from the working curve.

River Sediment

Mean recoveries of 17 elements from the standardized river sediment are presented in Tables 18 and 19. Mean recoveries were calculated by averaging results for both AAS and ICP and correcting (where possible) for the concentration of each element determined from the appropriate unspiked EP for each material. The concentration of element recovered was then compared to the original concentration added to determine the percent recovery. The raw data from which these summaries were derived are presented in Appendix G.

Blank spike recoveries at the 150%, 100%, and 50% levels for the pH 5 samples and at the 100% spike level for the low-pH samples are also included in Appendix G. Recoveries for most metals in the blank spikes were in the range 85% to 115% with the following exceptions:

TABLE 18. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSES OF STANDARDIZED RIVER SEDIMENT (pH 5)

	Mean recovery (%)[a]		
Element	Spike 1 (150%)	Spike 2 (100%)	Spike 3 (50%)
Al	3±2	3±1	3±1
Sb	<1	<2	<4
As	33±3	34±2	30±1
Ba	102±8	110±10	107±13
Be	10±2	13±1	<8
Cd	149±14	169±12	169±13
Cr	<1	<1	<3
Cu	21±3	15±2	8±4
Fe	2±1	2±0	2±0
Pb	34±3	28±1	17±2
Mn	115±8	121±5	138±5
Hg	<5	<10	<20
Ni	93±2	94±6	89±16
Se	17±2	21±1	9±2
Ag	<1	<1	<1
Tl	55±7	46±3	33±6
Zn	103±5	108±4	115±15

[a] Mean and standard deviation from triplicate analyses performed by AAS & ICP.

TABLE 19. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSES OF STANDARDIZED RIVER SEDIMENT (LOW pH)

	Mean recovery (%)[a]		
Element	Spike 1(150%)	Spike 2(100%)	Spike 3(50%)
Al	96 ± 28	68 ± 31	323 ± 39
Sb	15 ± 1.7	13 ± 0.8	11 ± 0.2
As	20 ± 9	56 ± 16	39 ± 18
Ba	95 + 5	98 ± 3	89 ± 4
Be	105 + 5	100 ± 0	80 ± 0
Cd	102 ± 24	110 ± 30	187 ± 47
Cr	104 ± 1	101 ± 2	85 ± 2
Cu	111 ± 9	117 ± 9	112 ± 9
Fe	134 ± 12	137 ± 13	139 ± 13
Pb	98 ± 11	94 ± 12	62 ± 13
Mn	98 ± 4	96 ± 5	125 ± 9
Hg	55 ± 5	89 ± 4	138 ± 20
Ni	131 ± 19	155 ± 42	191 ± 89
Se	9 ± 0	<8	<13
Ag	1.3 ± 0.1	1.5 ± 0.4	1.8 ± 0.2
Tl	87 ± 5	78 ± 3	65 ± 7
Zn	96 ± 3	96 ± 6	78 ± 4

---

[a] Mean and standard deviation from triplicate analyses performed by AAS and ICP.

- Ag recoveries were <10% for all blank spikes. Ag is unstable in the mixed standard solutions which contained HCl.

- Hg recoveries were consistently high. Hg spike levels were quite low (0.5 to 2 ug/L) and background and/or contamination is likely.

- Cd recoveries were high for the 50% pH 5 (150%) and 100% low-pH (130%) blank spikes but were low for the 100% pH 5 (73%) samples and normal (96%) for the 150% pH 5 sample. The lower two spike levels were 20 and 30 ug/L and contamination and/or poor precision is possible at these levels.

- Ni recovery from the 50% pH 5 spike was 67%. This is difficult to explain since recoveries of Ni from the EP extracts were all near 100%.

For the pH 5 adjusted river sediment samples, recoveries ranged from <1% to 169%; for the low-pH samples, the recoveries ranged from 1% to 323%. In general, recoveries of the high (150%) spikes were higher than for the low (50%) spikes. The most obvious difference is that the recoveries for the low-pH samples are much higher and much closer to 100% than the pH 5 recoveries. Several mechanisms are operative:

- The river sediment was dried and finely ground resulting in a high surface area per gram. Chemical or physical adsorption of the metal ions could occur as well as other "ion exchange" mechanisms. These would be less likely in highly acidic samples.

- Precipitation or co-precipitation as hydroxides, sulfates, phosphates etc. would be more likely at pH 5 than in the low-pH solutions. The concentrations of Fe, Al, Mn, Cu, and Zn in the bulk solid were high (Table 17) and exceeding the solubilities of $Fe(OH)_3$, $Cr(OH)_3$, $Al(OH)_3$, AgCl, and other compounds is quite likely.

- Spikes were made at inappropriate concentrations either too low for precise detection in the matrix (Cd, Hg) at the 50% spike level or too high due to the fact that spike levels were based upon the total metal content not on unspiked extract levels.

- For the low-pH extractions, Fe, Cd, Al, Hg, and Ni had at least one spike recovery >130%. The Hg and Cd spikes were at low levels (higher error) while Fe, Al, and Ni were at high concentrations in the sediment and the high recoveries probably reflect the differences in extraction efficiency caused by the slight differences in pH between the unspiked and spiked sediment samples.

The recoveries of Se, Sb, and As were poor for both low-pH and pH 5 extracts. These elements would most likely be present as oxyanions and would not precipitate as hydroxides. The most likely mechanisms of loss of these would be adsorption or co-precipitation.

Fly Ash

The fly ash samples were dried, ground, and homogenized prior to bulk analysis and Phase 3 extractions. Bulk concentrations of the elements of interest were determined by ERCO and verified by an outside laboratory. The ERCO values are presented in Table 17.

Each sample was prepared for the EP extraction in the same manner that was used for river sediment and according to methods outlined by EPA[1]. After addition of distilled water to the dried ash, the samples were spiked in triplicate at these different concentration levels representing 150%, 100%, and 50% of the total metal concentrations determined from the bulk analysis. The sample pH was either maintained at its initial level during spike addition by concurrent titration with 0.25 N Na_2CO_3 or was allowed to remain at a low pH due to the acid added while spiking. For each spike level, a "blank spike" was prepared by adding the spikes to distilled water. These solutions were used to verify the integrity of the sample spikes.

Mean recoveries of 17 elements from the standardized fly ash are presented in Table 20 and 21. Mean recoveries were calculated by averaging results for both AAS and ICP and correcting (where possible) for the concentration of each element determined from an unspiked EP for each material. The concentration of element recovered was then compared to the original concentration added to determine the percent recovery.

The raw data from which the mean recoveries were calculated, as well as the recoveries for the "blank spike" solutions are presented in Appendix H.

Examination of the data for the "blank spikes" (Tables H-3 to H-6) shows that Ag is unstable in the mixed standard solution and that Hg levels were low enough to be severely affected by slight contamination. All other recoveries were in the range of 90% to 110% except for the 150% Ni recovery of 62% at pH 5. The 150% spike for Ni should have been added from a standard made from the stock solutions for both the 50% spike (0.50 mg/L) plus the 100% spike (0.90 mg/L) for an expected concentration in the 150% spike of 1.4 mg/L). If the 0.50 mg/L solution were inadver-

TABLE 20. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSES OF STANDARDIZED FLY ASH (pH 5)

Element	Mean recovery (%)[a] Spike 1 (150%)	Spike 2 (100%)	Spike 3 (50%)
Al	80±2	79±3	46±11
Sb	31±2	16±2	10±1
As	40±3	17±4	<1
Ba	7±1	5±1	9±1
Be	94±6	93±6	75±6
Cd	102±1	104±1	101±3
Cr	33±3	38±4	26±4
Cu	95±2	99±1	89±2
Fe	57±5	19±2	<1
Pb	<20	<29	<57
Mn	105±7	106±5	102±2
Hg	127±13	97±12	47±12
Ni	69±3	109±4	106±12
Se	44±9	19±2	<10
Ag	16±8	3±1	3±0
Tl	75±6	65±4	46±12
Zn	102±1	106±2	99±5

[a] Mean and standard deviation from triplicate analyses performed by AAS & ICP.

TABLE 21. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSIS OF STANDARDIZED FLY ASH (LOW pH)

	Mean recovery (%)[a]		
Element	Spike 1 (150%)	Spike 2 (100%)	Spike 3 (50%)
Al	110 ± 16	109 ± 24	69 ± 29
Sb	14 ± 4	15 ± 4	10 ± 1
As	75 ± 25	102 ± 30	13 ± 7
Ba	10 + 4	13 ± 6	21 ± 7
Be	93 + 13	90 ± 15	92 ± 7
Cd	97 ± 3	100 ± 6	100 ± 8
Cr	103 ± 6	104 ± 6	78 ± 9
Cu	100 ± 5	100 ± 6	92 ± 3
Fe	85 ± 13	129 ± 21	67 ± 19
Pb	<40	<57	NC
Mn	105 ± 8	93 ± 4	92 ± 1
Hg	115 ± 45	160 ± 10	154 ± 24
Ni	114 ± 14	114 ± 17	138 ± 58
Se	90 ± 39	95 ± 25	22 ± 0
Ag	1 ± 0.1	1 ± 0.3	1 ± 0
Tl	93 ± 4	93 ± 3	80 ± 15
Zn	96 ± 9	97 ± 7	44 ± 1

NC = Not calculated.

[a] Mean and standard deviation from triplicate analyses performed by AAS and ICP.

tently not added to the standard, the Ni recovery (based on a final expected concentration of 0.9 mg/L, not 1.4 mg/L) would be 96%.

As with the river sediment samples, recoveries from the low pH extracts were consistently better than from the pH 5 extracts and the recoveries of the anionic species (As, Sb, Se) were low at both pH values, Ni again exhibited high recoveries (particularly by AAS) due to Fe interference (7). In addition, the fly ash samples exhibited low recoveries for those elements with insoluble sulfates (Ba, Pb) as did the fly ash samples utilized in Phase 1.

EPA Municipal Digested Sludge

The sample of EPA municipal digested sludge was dried, ground, and homogenized prior to use for Phase 3 extractions and bulk analysis. Bulk concentrations of the elements of interest were determined by ERCO and verified by an outside laboratory. The bulk concentrations determined by ERCO are presented in Table 17. All values were within the published 95% confidence levels for the sludge except Zn (range 1190-1450 ug/g; found 1050 ug/g).

Each sample was prepared for the EP extraction according to methods outlined by EPA[1] and in the manner that was employed for the fly ash and river sediment samples. Mean recoveries of 17 elements from the sludge are presented in Tables 22 and 23. Mean recoveries were calculated by averaging results for both AAS and ICP and correcting (where possible) for the concentration of each element determined from the appropriate unspiked EP for each material. The concentration of element recovered was then compared to the original concentration added to determine the percent recovery.

TABLE 22. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSES OF EPA MUNICIPAL DIGESTED SLUDGE (pH 5)

	Mean recovery (%)[a]		
Element	Spike 1 (150%)	Spike 2 (100%)	Spike 3 (50%)
Al	1±0	1±0	2±1
Sb	8±1	7±2	7±1
As	<42	<80	NC
Ba	9±2	7±2	4±1
Be	<3	<4	<8
Cd	30±4	24±4	26±5
Cr	5±1	5±1	6±1
Cu	8±1	6±0	6±1
Fe	<1	<1	2+1
Pb	<1	<1	<2
Mn	63±6	58±3	60±5
Hg	183±23	390±35	913±153
Ni	53±6	47±5	48±6
Se	<8	<13	<25
Ag	<2	<1	2
Tl	26±5	<27	<53
Zn	33±3	29±3	31±3

NC = Not calculated.

[a] Mean and standard deviation from triplicate analyses performed by AAS & ICP.

TABLE 23. SUMMARY OF SPIKE RECOVERIES FROM PHASE 3 ANALYSES OF EPA MUNICIPAL DIGESTED SLUDGE (LOW pH)

	Mean recovery (%)[a]		
Element	Spike 1 (150%)	Spike 2 (100%)	Spike 3 (50%)
Al	473 ± 53	83 ± 6	1040 ± 260
Sb	16 ± 3	19 ± 5	<6
As	84 ± 3	62 ± 5	155 ± 61
Ba	7.5 ± 1	16 ± 6	30 ± 4
Be	NC	NC	NC
Cd	205 ± 31	208 ± 8	220 ± 5
Cr	198 ± 7	154 ± 6	213 ± 14
Cu	203 ± 14	340 ± 44	272 ± 28
Fe	362 ± 68	213 ± 15	88 ± 55
Pb	2.9 ± 0.4	<2	27 ± 11
Mn	203 ± 8	191 ± 1	240 ± 18
Hg	41 ± 5	110 ± 10	162 ± 26
Ni	271 ± 3	317 ± 128	283 ± 225
Se	<8	<13	<27
Ag	1.7 ± 0.1	<1.2	<2.4
Tl	175 ± 94	168 ± 128	232 ± 165
Zn	200 ± 23	181 ± 31	292 ± 38

NC = Not calculated.

[a] Mean and standard deviation from triplicate analyses performed by AAS and ICP.

The raw data from which the summary recoveries were calculated as well as the "blank spike" recoveries are in Appendix I.

Recoveries of the metals in the blank spikes were all between 90% and 110% with the exception of Ag which is unstable in the mixed standards and Hg which was spiked at low levels.

Recoveries of the metals in the extracts were extremely poor. For the pH 5 extracts, no element exhibited recoveries in the 80% to 120% range. For the low pH extracts only four of the 51 recoveries were in the 80% to 120% range. Recoveries in the pH 5 extracts were all extremely low (except Hg) while the low pH recoveries were highly erratic. For the low pH extracts, Sb, Pb, Ba, Se, and Ag exhibited low recoveries. The Ag was expected to be low since the spike solution contains chloride and precipitates the Ag. The digested sludge apparently contains sulfate which would selectively precipitate Pb and Ba. The Sb and Se recoveries, as with the fly ash and river sediment, were low for both the low pH and pH 5 extracts.

Comparison of AAS and ICP Recoveries

While the intent of Phase 3 was to determine the ability of the EP test to leach spikes from standard reference materials, the data are also useful in observing the comparative performance of AA and ICP. Both techniques were used to analyze the same undigested EP leachates with the same spike levels and consequently comparison of their performance is straightforward. The data are contained in Tables G-1, G-2, H-1, H-2, I-1, and I-2.

River Sediment

For the river sediment extracts at low pH, the following obvious differences attributable to the techniques were found:

- For As, although recoveries by both techniques were poor, ICP recoveries were significantly higher than AAS, probably due to a loss of As in the furnace AAS analysis which cannot occur in the ICP analysis.

- For Ni, the AAS recoveries were extremely high (149% to 271%) while the ICP recoveries were more reasonable (110% to 118%). This may be due to a positive interference of Fe on the AAS analysis (7).

- For the other analyses the two techniques performed comparably.

The pH 5 river sediment extracts showed the following technique-related differences:

- For As, the ICP detection limits were much higher than the AAS limits and much higher than the ICP limits in the lower pH samples.

- For Ba, the AAS recoveries were consistently higher than 100% while the ICP recoveries were consistently near 100% probably due to an interference in the AAS analysis. Both spectral and ionization interferences are known for Ba analysis by AAS (7).

- Ni recoveries by ICP were nearer to 100% than the lower AAS recoveries probably due to a chemical interference, e.g., the higher temperature ICP was capable of breaking apart molecular species or occluded clusters (suspended fine precipitates) which the AAS was not. The AAS recoveries were low even though an enhancement of the Ni signal by Fe was probably occurring.

- Zn recoveries by both techniques were high. However, the AAS recoveries were significantly higher and less precise than those of ICP.

Fly ash

The low-pH fly ash data (Table H-1) show the following differences:

- As recoveries were generally low and variable for both techniques with the ICP recoveries higher than AAS.
- Be recoveries were near 100% for ICP, near 80% for AAS.
- For Cr, the ICP recoveries at the high and medium levels were 100% while the corresponding AAS values were 108%. At the low spike (1.25 mg/L) the AAS recovery (86%) was better than the ICP (71%).
- For Cu, the AAS recoveries averaged 93% while the ICP recoveries averaged 101%. A slight suppression of Cu signals by Fe has been reported for flame AAS (7).
- For Ni, again the AAS recoveries are well above 100% while the ICP recoveries were 102%. The Fe interference on nickel analysis by AAS is the likely cause.
- For Se at the low levels used for all spikes, ICP detected no Se while at least for the medium and high spikes the AAS recoveries were calculable and above 90%.
- For Zn, at the high and medium spike levels, AAS recoveries (91% and 89%) were lower than ICP (100% for both).

The low spike recoveries were poor for both AAS (43%) and ICP (45%).

– The pH 5 extracts of fly ash (Table H-2) yielded comparable recoveries by both techniques with two exceptions:

- For Be, the ICP recoveries were higher (and closer to 100%) than the AAS recoveries (ICP; 99%, 98%, 79% - AAS; 90%, 88%, 71%).
- For Mn, the AAS recoveries (111%, 111%, 104%) were higher than the ICP recoveries (99%, 102%, 100%).

EPA Municipal Digested Sludge

Recoveries in both pH 5 and the low-pH extracts were erratic by both techniques and any definite conclusions as to technique related differences would be suspect.

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6. M. Ihnat, Anal. Chim. Acta, 82, 293, 1976.

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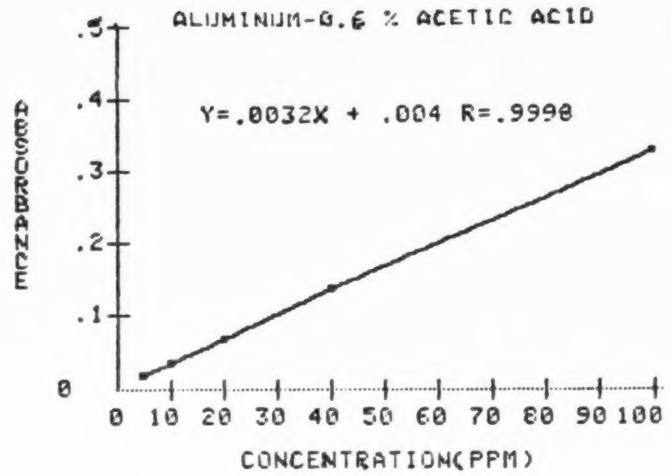
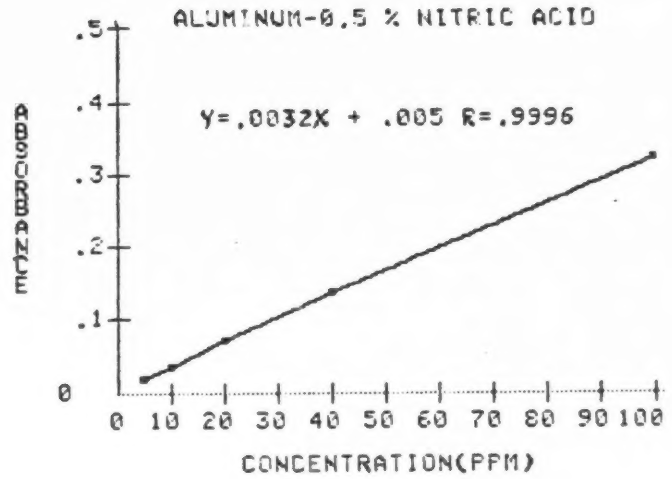
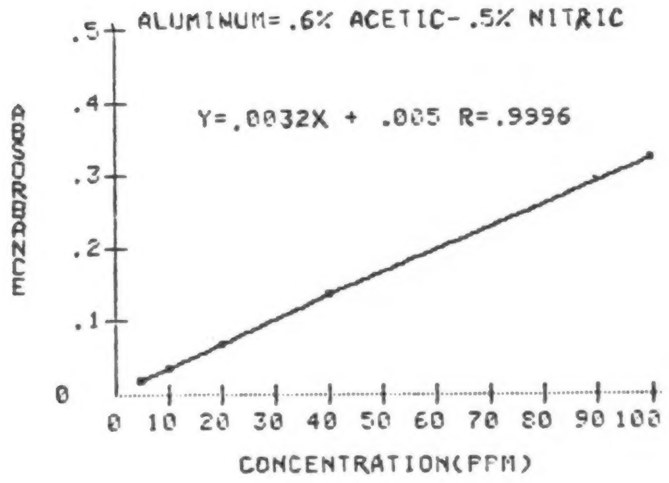
APPENDIX A

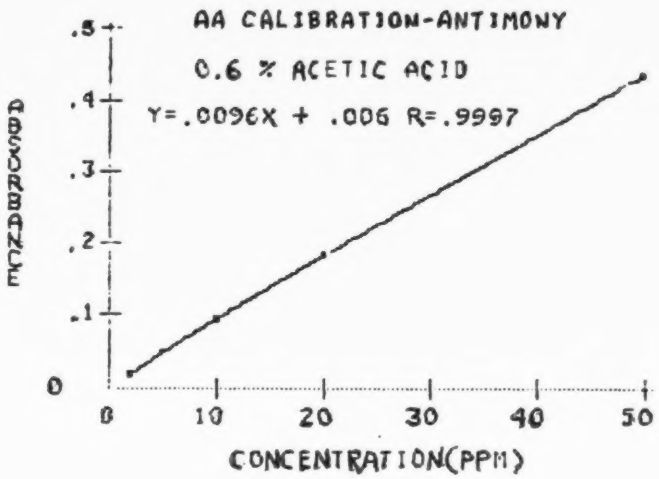
RAW DATA FROM CALIBRATION STUDY

This appendix summarizes results obtained for the calibration study conducted to determine the effects of three matrices (0.6% acetic acid, 0.5% nitric acid and 0.6% acetic acid/0.5% nitric acid) on measurements made by atomic absorption spectroscopy (AAS) and inductively coupled argon plasma emission spectroscopy (ICP). Results are presented for 17 elements for AAS (Al, Sb, As, Ba, Be, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Se, Ag, Tl, and Zn) and for 16 elements for ICP (Al, Sb, As, Ba, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Se, Ag, Tl, and Zn).


ALUMINUM-0.6 % ACETIC ACID
Y=.0032X + .004 R=.9998
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)


ALUMINUM-0.5 % NITRIC ACID
Y=.0032X + .005 R=.9996
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)


ALUMINUM=.6% ACETIC-.5% NITRIC
Y=.0032X + .005 R=.9996
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)


AA CALIBRATION-ANTIMONY
0.6 % ACETIC ACID
Y=.0096X + .006 R=.9997
ABSORBANCE
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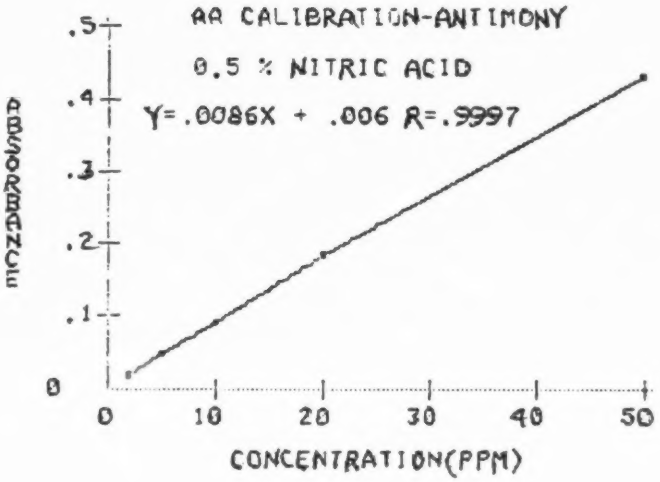
.4
.3
.2
.1
0
0
10
20
30
40
50
CONCENTRATION(PPM)


AA CALIBRATION-ANTIMONY
0.5 % NITRIC ACID
Y=.0086X + .006 R=.9997
ABSORBANCE
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.3
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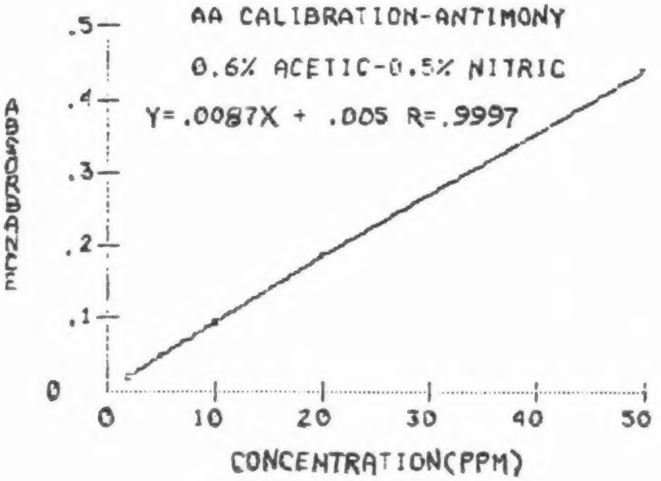
30
40
50
CONCENTRATION(PPM)


AA CALIBRATION-ANTIMONY
0.6% ACETIC-0.5% NITRIC
Y=.0087X + .005 R=.9997
ABSORBANCE
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.4
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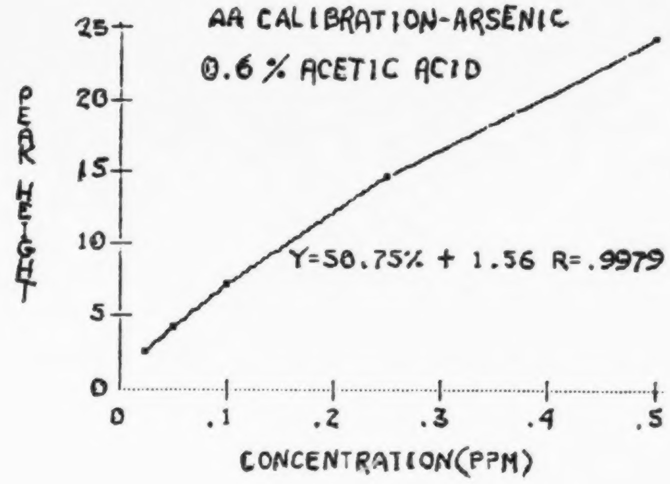
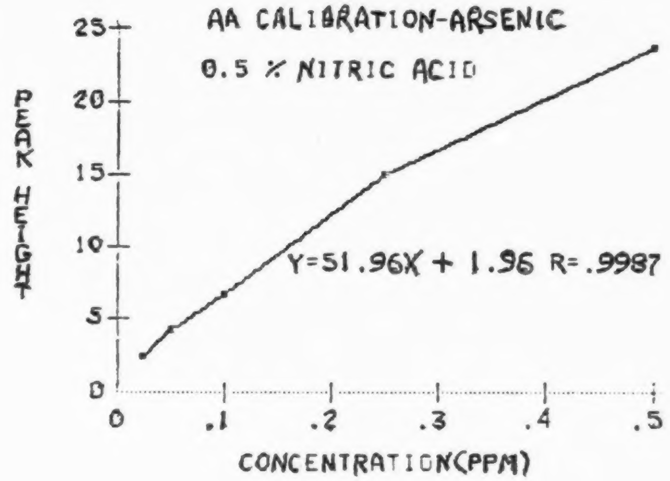
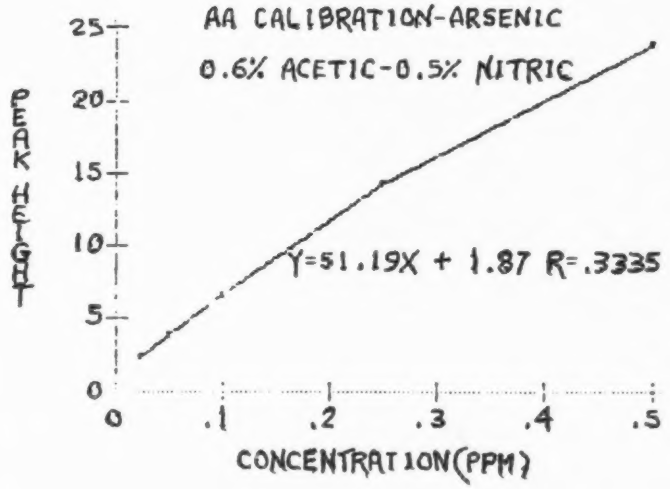
30
40
50
CONCENTRATION(PPM)


AA CALIBRATION-ARSENIC
0.6 % ACETIC ACID
PEAK HEIGHT
25
20
15
10
5
0
Y=50.75% + 1.56 R=.9979
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)


AA CALIBRATION-ARSENIC
0.5 % NITRIC ACID
PEAK HEIGHT
25
20
15
10
5
0
Y=51.96X + 1.96 R=.9987
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)


AA CALIBRATION-ARSENIC
0.6% ACETIC-0.5% NITRIC
PEAK HEIGHT
25
20
15
10
5
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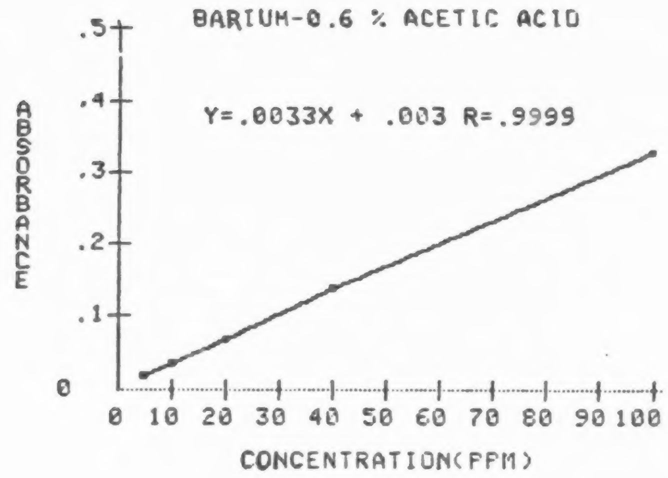
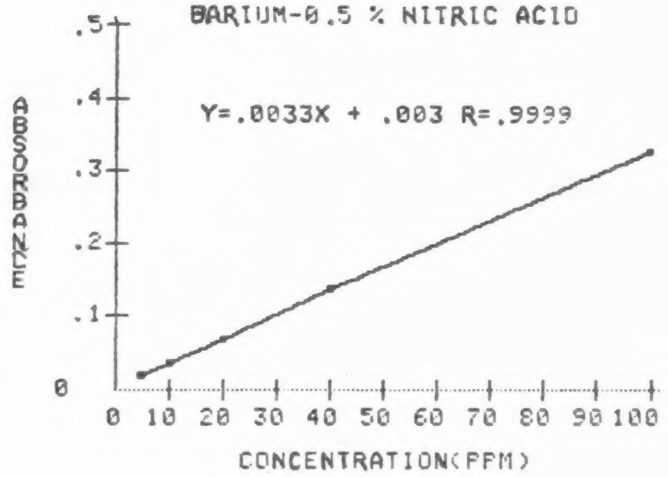
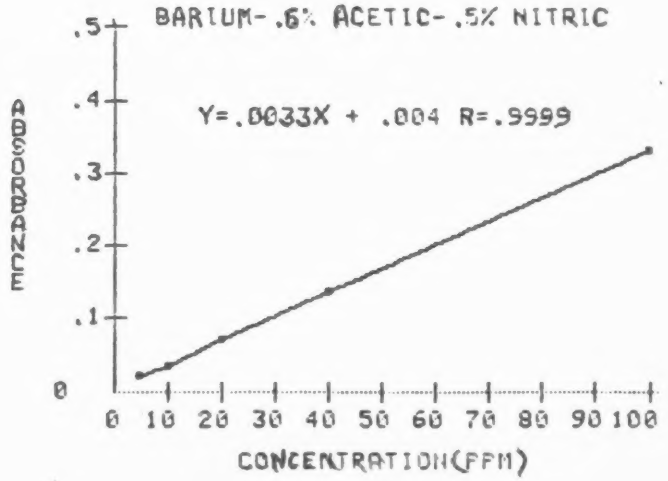
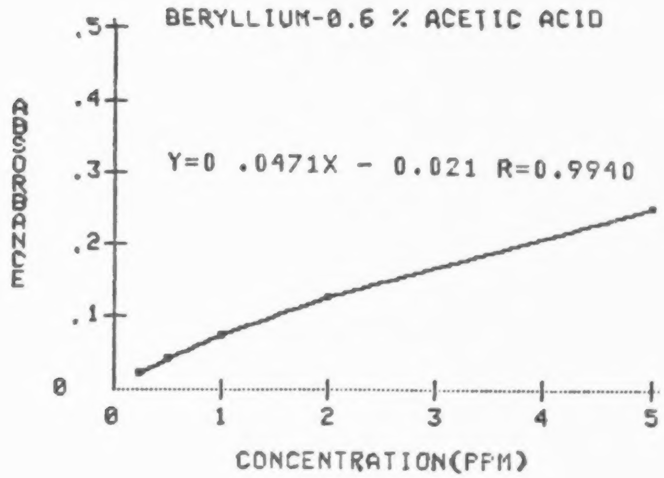
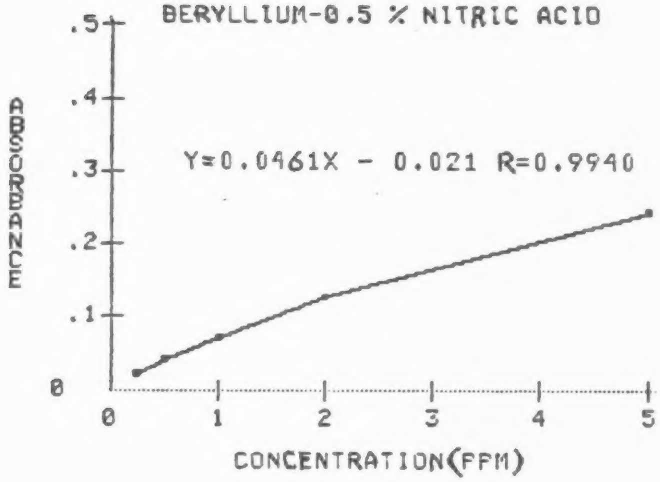
Y=51.19X + 1.87 R=.3335
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)

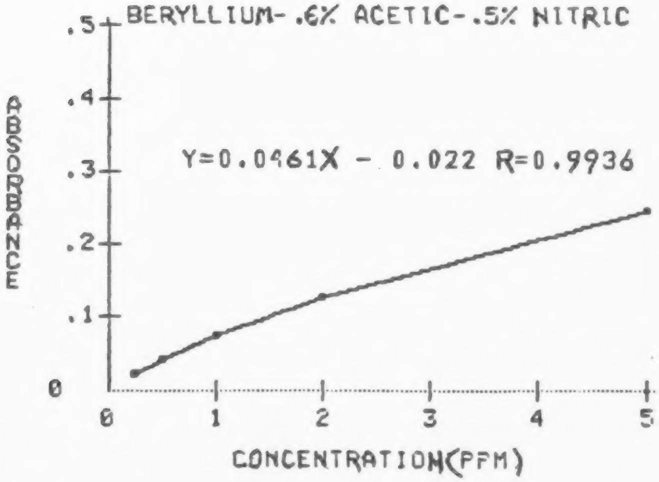

BARIUM-0.6 % ACETIC ACID
Y=.0033X + .003 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)

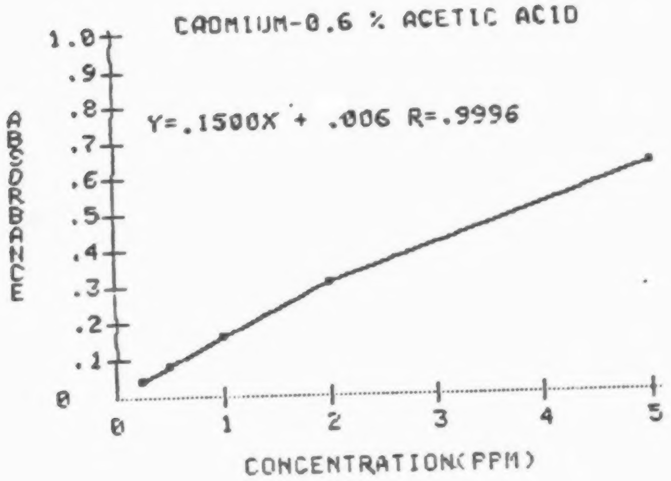

BARIUM-0.5 % NITRIC ACID
Y=.0033X + .003 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)

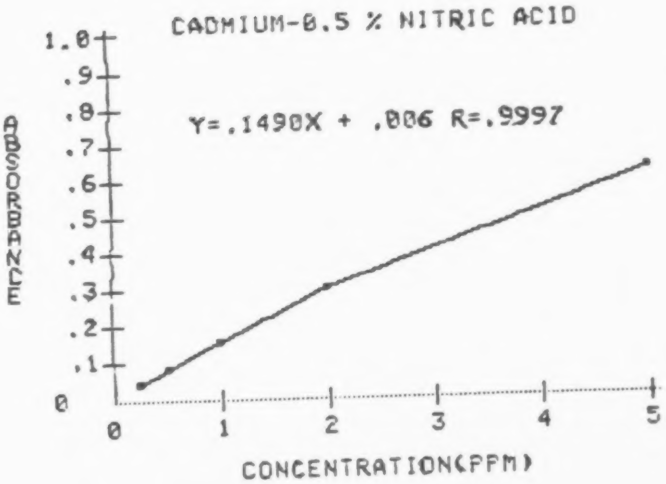

BARIUM-.6% ACETIC-.5% NITRIC
Y=.0033X + .004 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 10 20 30 40 50 60 70 80 90 100
CONCENTRATION(PPM)

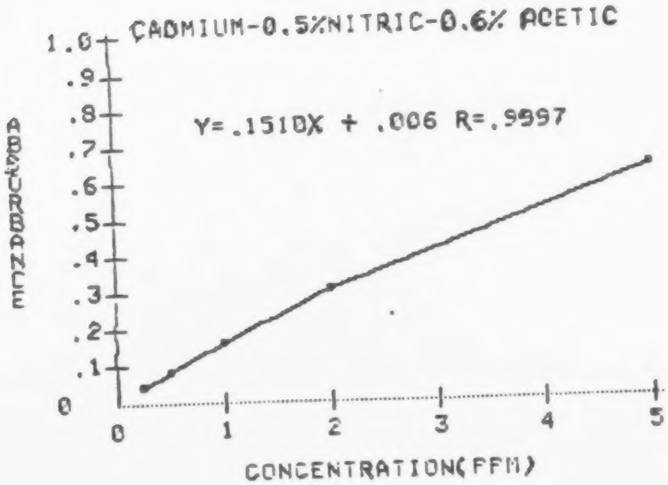

BERYLLIUM-0.6 % ACETIC ACID
ABSORBANCE
.5
.4
.3
.2
.1
0
Y=0 .0471X - 0.021 R=0.9940
0
1
2
3
4
5
CONCENTRATION(PPM)

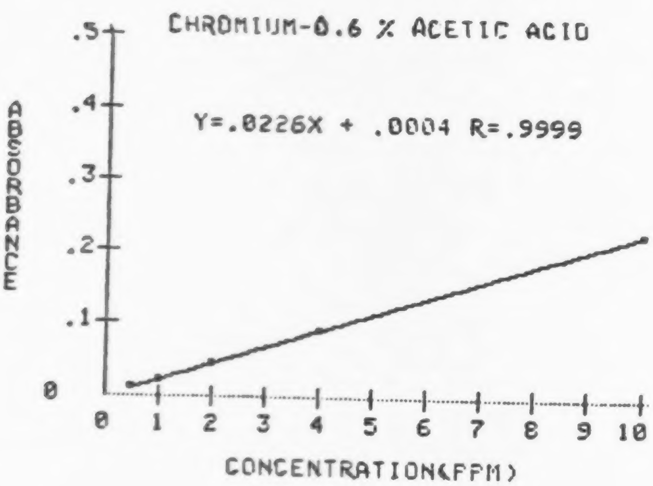

BERYLLIUM-0.5 % NITRIC ACID
ABSORBANCE
.5
.4
.3
.2
.1
0
Y=0.0461X - 0.021 R=0.9940
0
1
2
3
4
5
CONCENTRATION(PPM)

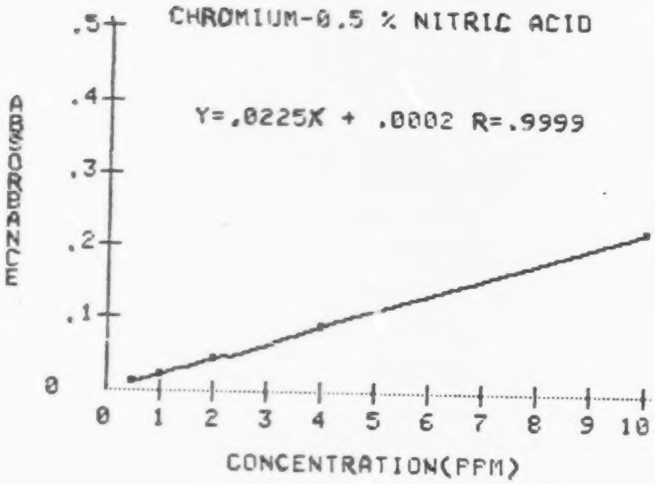

BERYLLIUM-.6% ACETIC-.5% NITRIC
ABSORBANCE
.5
.4
.3
.2
.1
0
Y=0.0461X - 0.022 R=0.9936
0
1
2
3
4
5
CONCENTRATION(PPM)

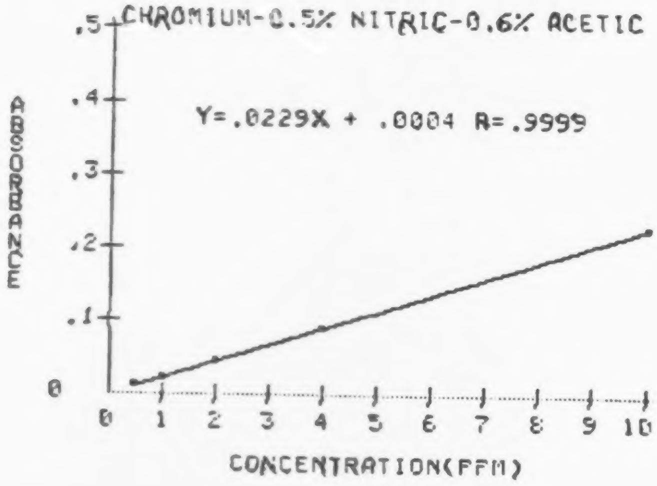

CADMIUM-0.6 % ACETIC ACID
Y=.1500X + .006 R=.9996
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

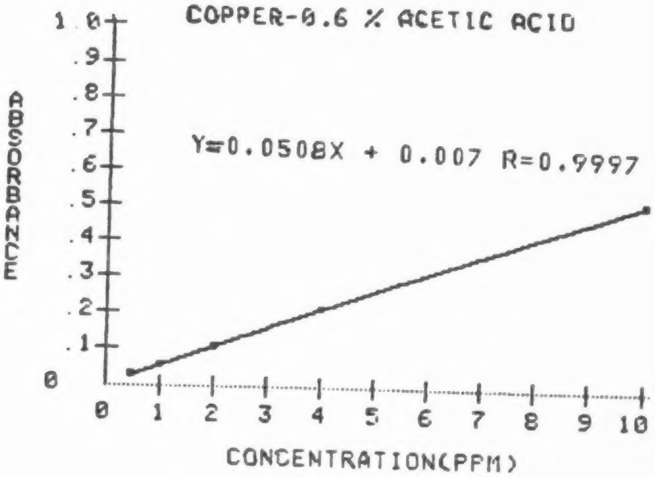

CADMIUM-0.5 % NITRIC ACID
Y=.1490X + .006 R=.9997
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

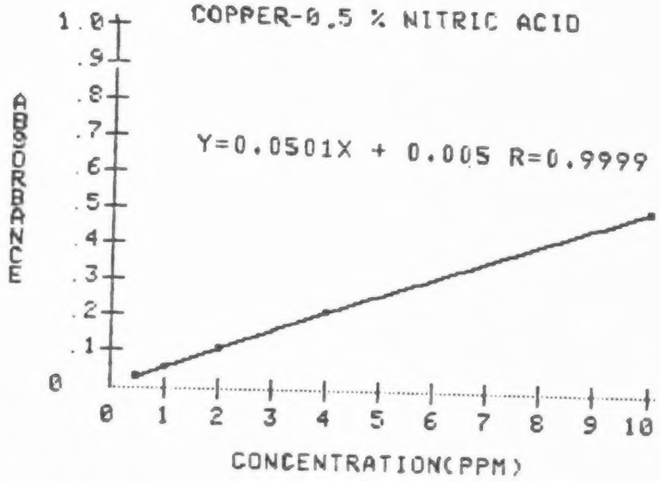

CADMIUM-0.5%NITRIC-0.6% ACETIC
Y=.1510X + .006 R=.9997
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

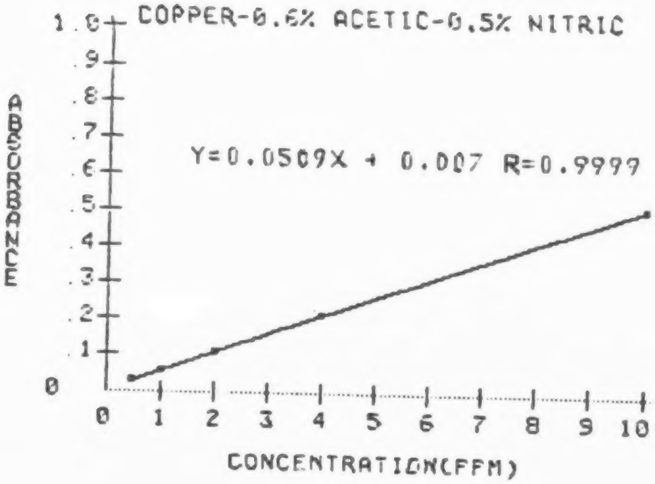

CHROMIUM-0.6 % ACETIC ACID
Y=.0226X + .0004 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

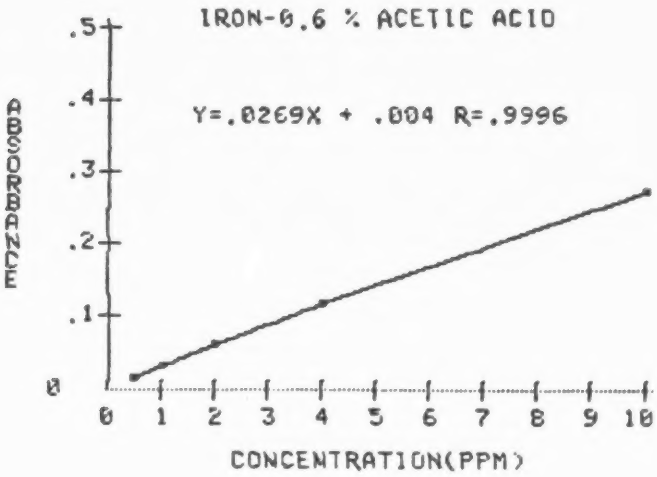

CHROMIUM-0.5 % NITRIC ACID
Y=.0225X + .0002 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

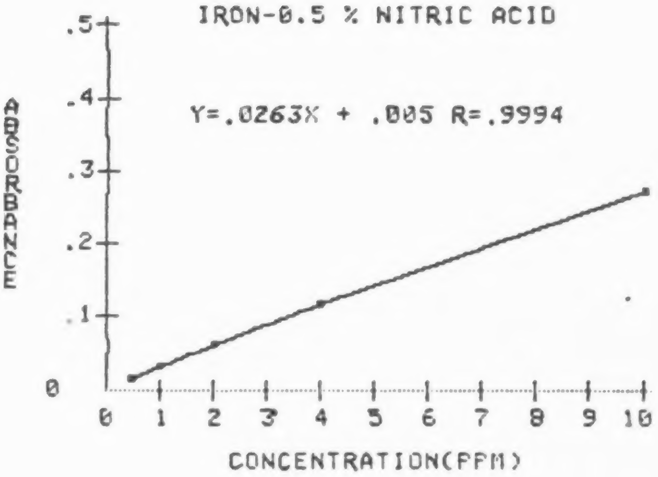

CHROMIUM-0.5% NITRIC-0.6% ACETIC
Y=.0229X + .0004 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

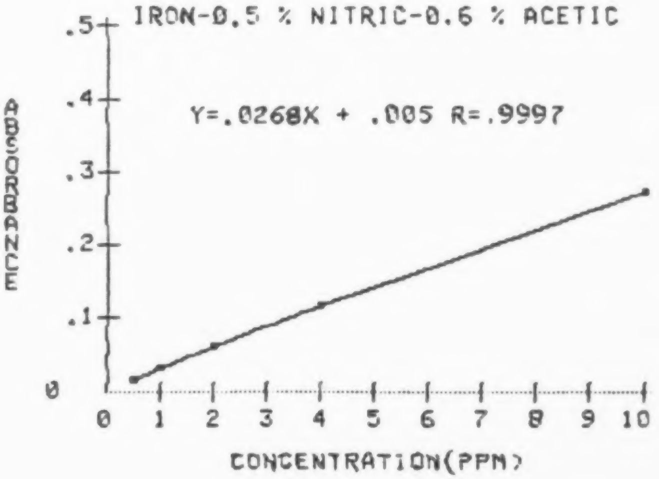

COPPER-0.6 % ACETIC ACID
Y=0.0508X + 0.007 R=0.9997
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
6
7
8
9
10
CONCENTRATION(PPM)

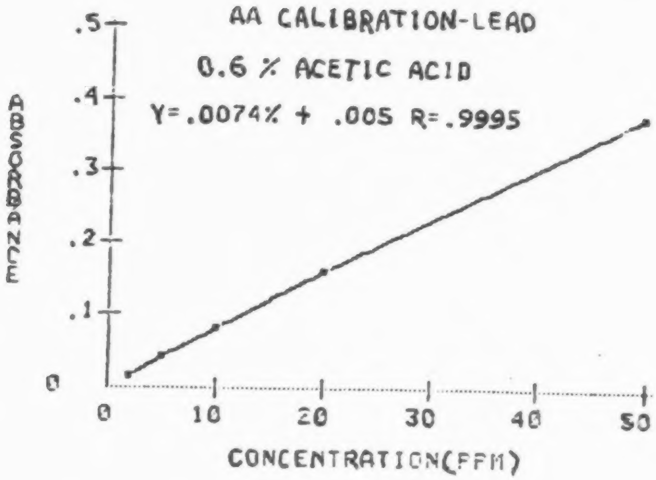

COPPER-0.5 % NITRIC ACID
Y=0.0501X + 0.005 R=0.9999
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
6
7
8
9
10
CONCENTRATION(PPM)

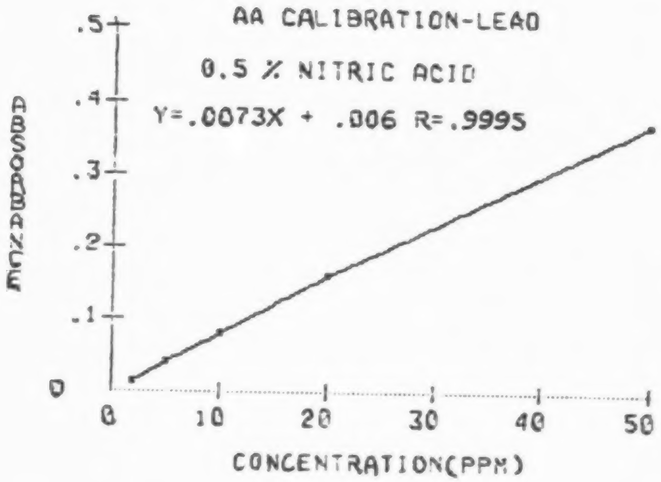

COPPER-0.6% ACETIC-0.5% NITRIC
Y=0.0509X + 0.007 R=0.9999
ABSORBANCE
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
6
7
8
9
10
CONCENTRATION(PPM)

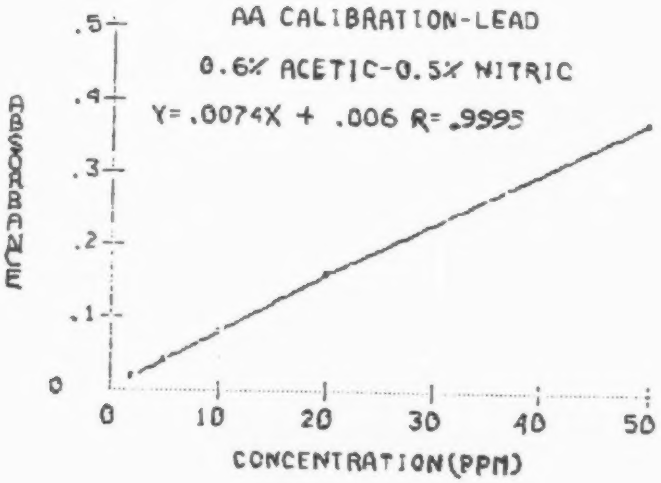

IRON-0.6 % ACETIC ACID
Y=.0269X + .004 R=.9996
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

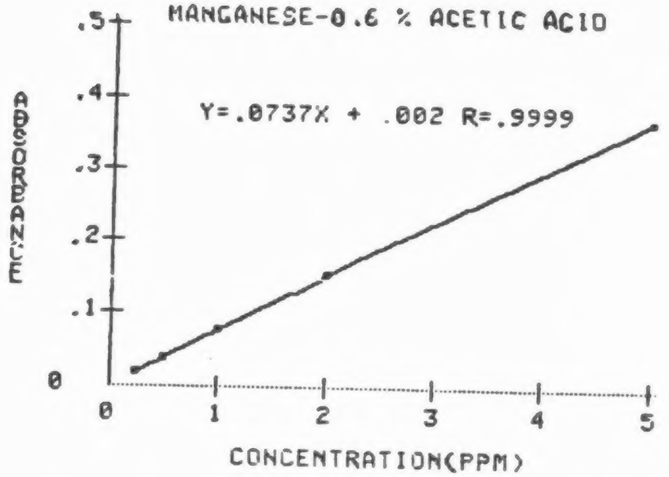

IRON-0.5 % NITRIC ACID
Y=.0263X + .005 R=.9994
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

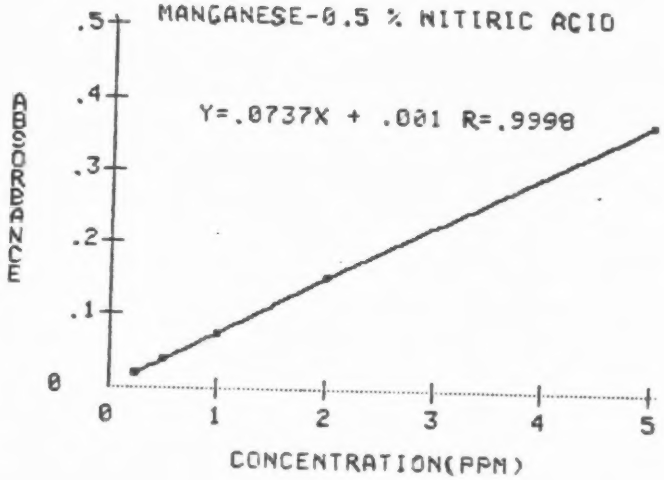

IRON-0.5 % NITRIC-0.6 % ACETIC
Y=.0268X + .005 R=.9997
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

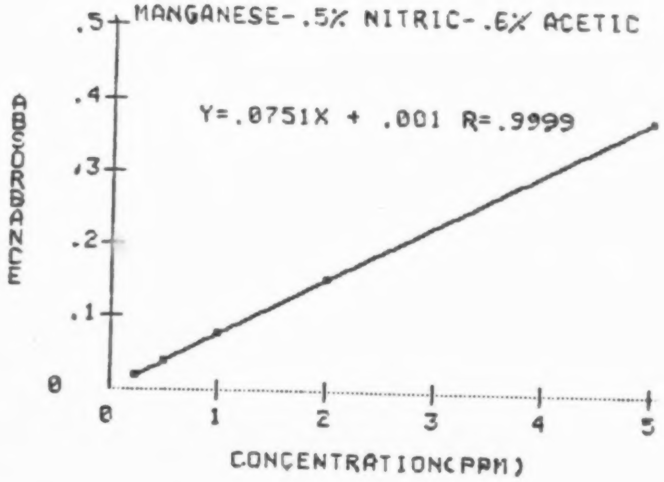

AA CALIBRATION-LEAD
0.6 % ACETIC ACID
Y=.0074% + .005 R=.9995
ABSORBANCE
.5
.4
.3
.2
.1
0
0
10
20
30
40
50
CONCENTRATION(PPM)

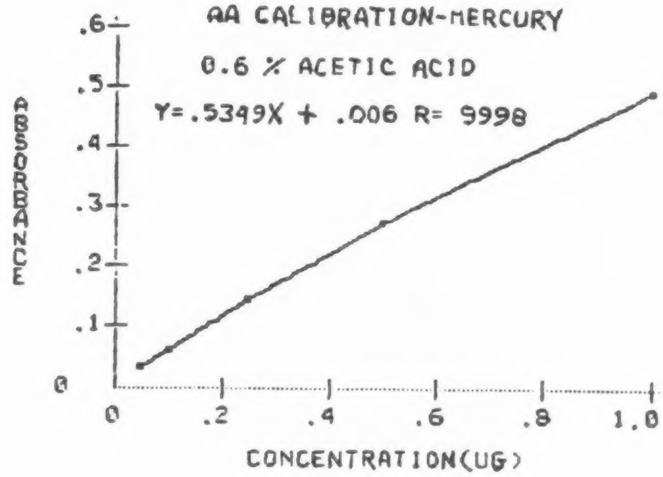

AA CALIBRATION-LEAD
0.5 % NITRIC ACID
Y=.0073X + .006 R=.9995
ABSORBANCE
.5
.4
.3
.2
.1
0
0
10
20
30
40
50
CONCENTRATION(PPM)

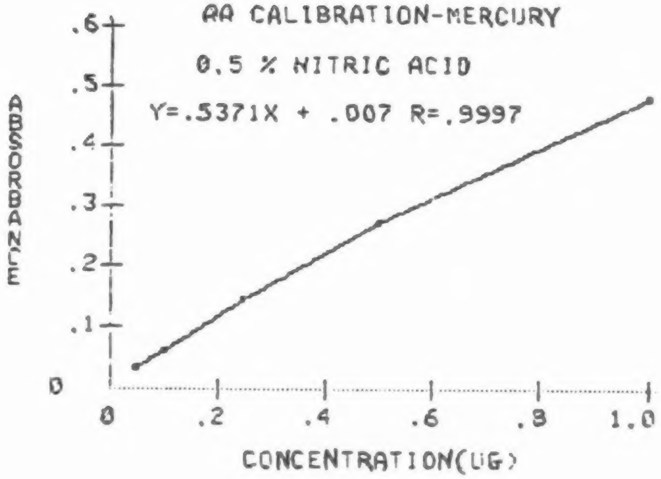

AA CALIBRATION-LEAD
0.6% ACETIC-0.5% NITRIC
Y=.0074X + .006 R=.9995
ABSORBANCE
.5
.4
.3
.2
.1
0
0
10
20
30
40
50
CONCENTRATION(PPM)

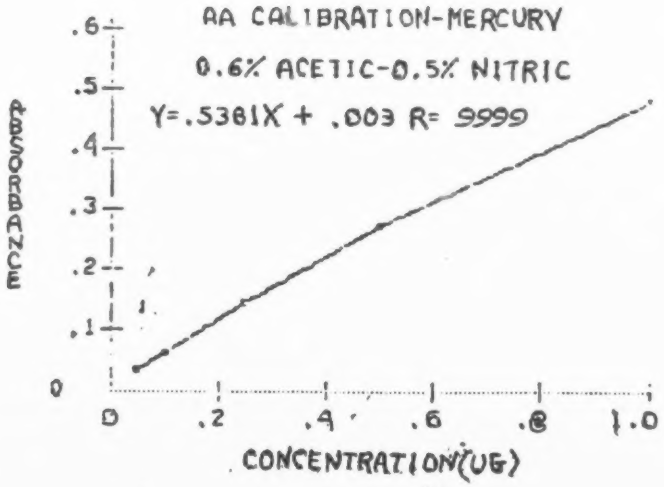

MANGANESE-0.6 % ACETIC ACID
Y=.0737X + .002 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

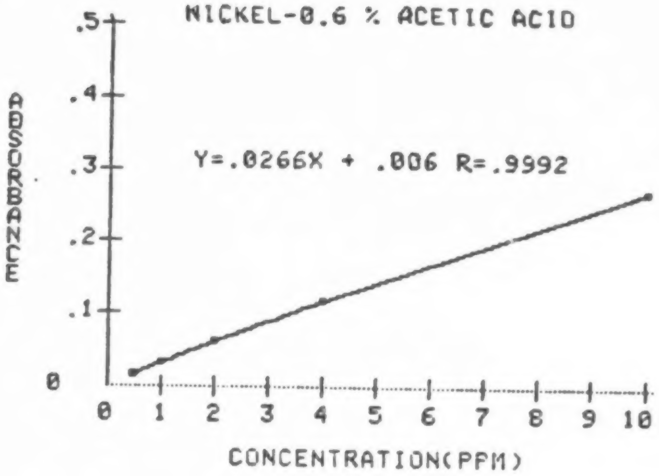

MANGANESE-0.5 % NITIRIC ACID
Y=.0737X + .001 R=.9998
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

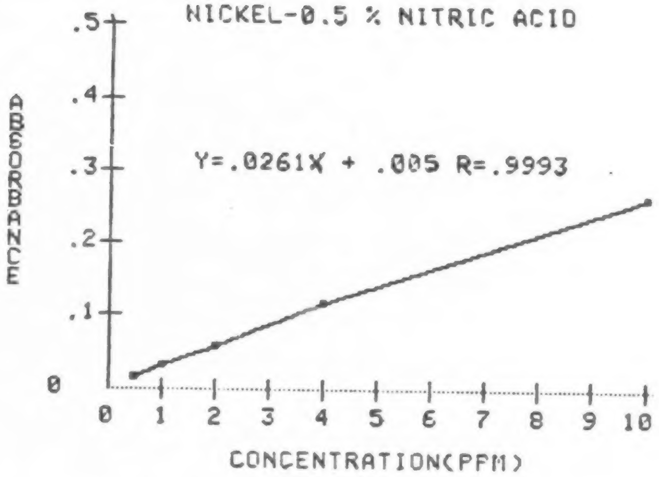

MANGANESE-.5% NITRIC-.6% ACETIC
Y=.0751X + .001 R=.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

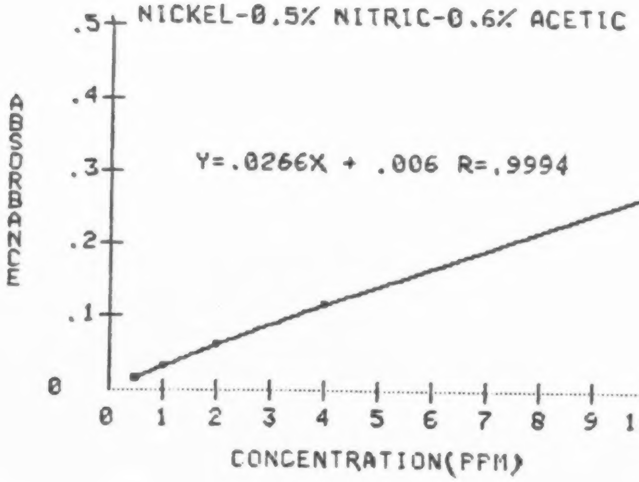

AA CALIBRATION-MERCURY
0.6 % ACETIC ACID
Y=.5349X + .006 R= 9998
ABSORBANCE
.6
.5
.4
.3
.2
.1
0
0
.2
.4
.6
.8
1.0
CONCENTRATION(UG)

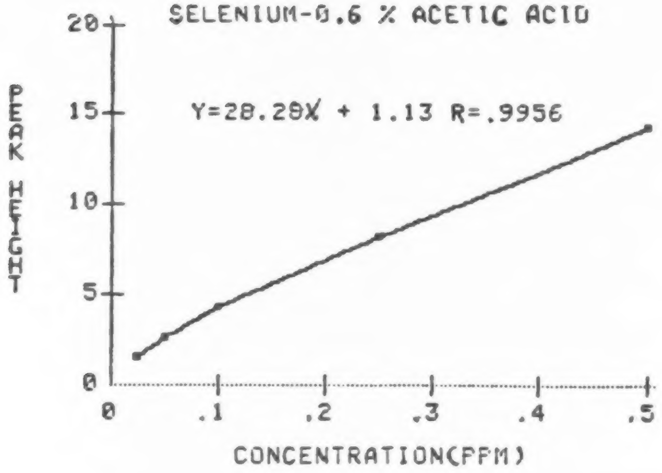

AA CALIBRATION-MERCURY
0.5 % NITRIC ACID
Y=.5371X + .007 R=.9997
ABSORBANCE
.6
.5
.4
.3
.2
.1
0
0
.2
.4
.6
.8
1.0
CONCENTRATION(UG)

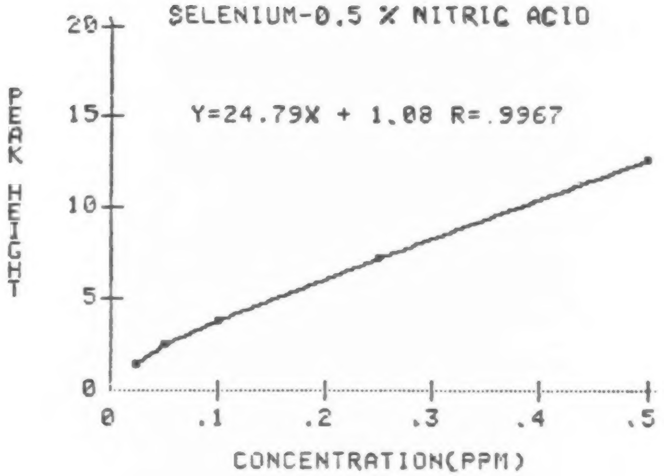

AA CALIBRATION-MERCURY
0.6% ACETIC-0.5% NITRIC
Y=.5381X + .003 R= 9999
ABSORBANCE
.6
.5
.4
.3
.2
.1
0
0
.2
.4
.6
.8
1.0
CONCENTRATION(UG)

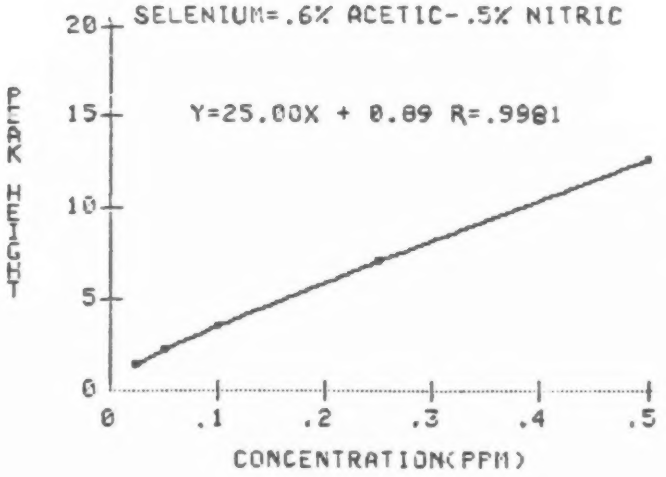

NICKEL-0.6 % ACETIC ACID
Y=.0266X + .006 R=.9992
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

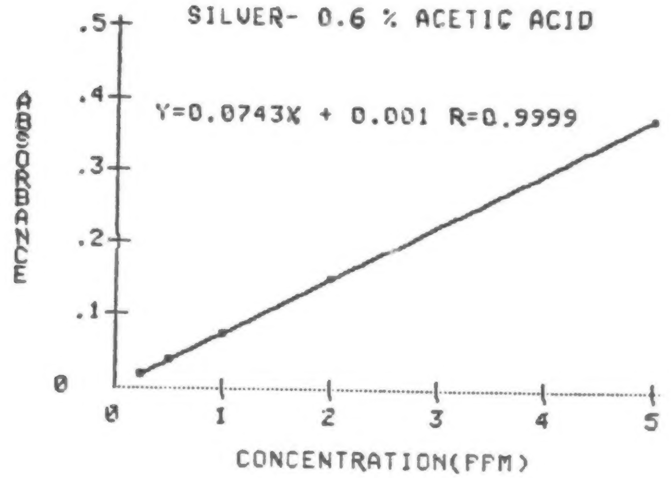

NICKEL-0.5 % NITRIC ACID
Y=.0261X + .005 R=.9993
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

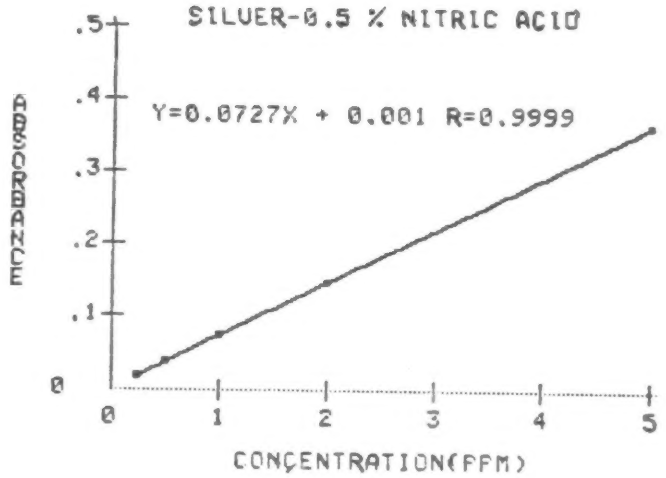

NICKEL-0.5% NITRIC-0.6% ACETIC
Y=.0266X + .006 R=.9994
ABSORBANCE
.5
.4
.3
.2
.1
0
0 1 2 3 4 5 6 7 8 9 10
CONCENTRATION(PPM)

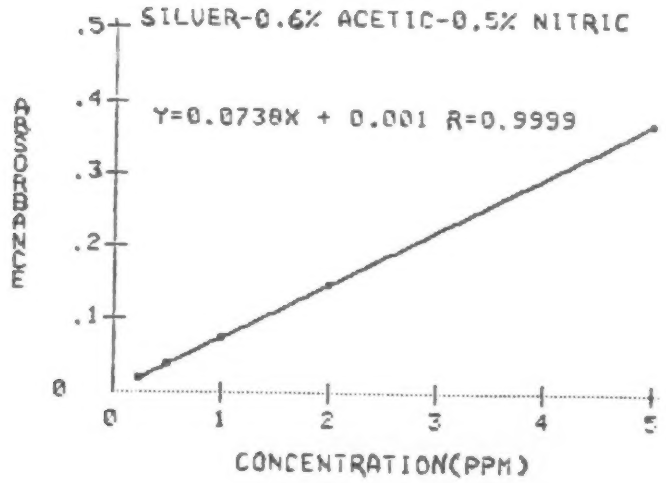

SELENIUM-0.6 % ACETIC ACID
Y=28.28X + 1.13 R=.9956
PEAK HEIGHT
20
15
10
5
0
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)

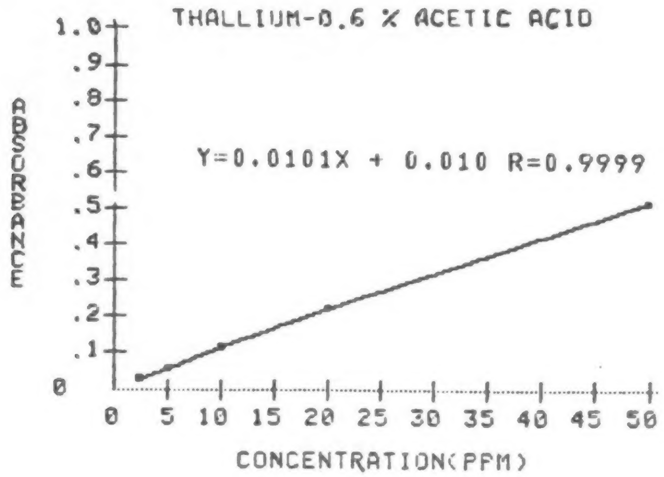

SELENIUM-0.5 % NITRIC ACID
Y=24.79X + 1.08 R=.9967
PEAK HEIGHT
20
15
10
5
0
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)

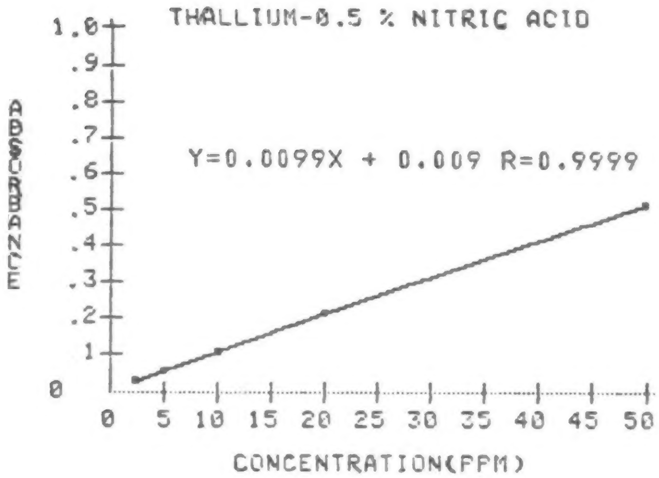

SELENIUM=.6% ACETIC-.5% NITRIC
Y=25.00X + 0.89 R=.9981
PEAK HEIGHT
20
15
10
5
0
0
.1
.2
.3
.4
.5
CONCENTRATION(PPM)

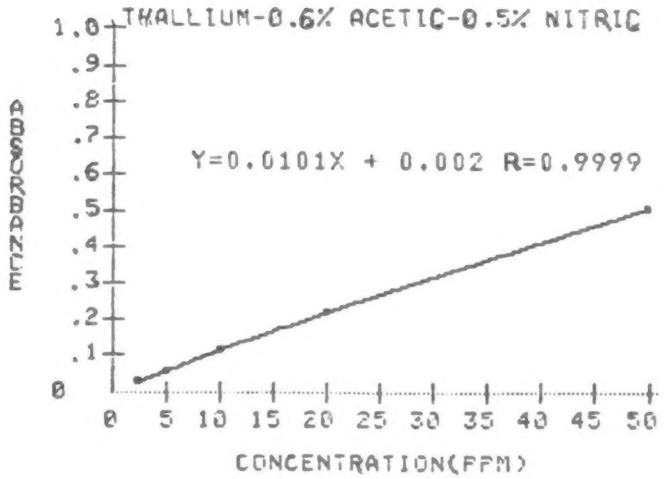

SILVER- 0.6 % ACETIC ACID
Y=0.0743X + 0.001 R=0.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

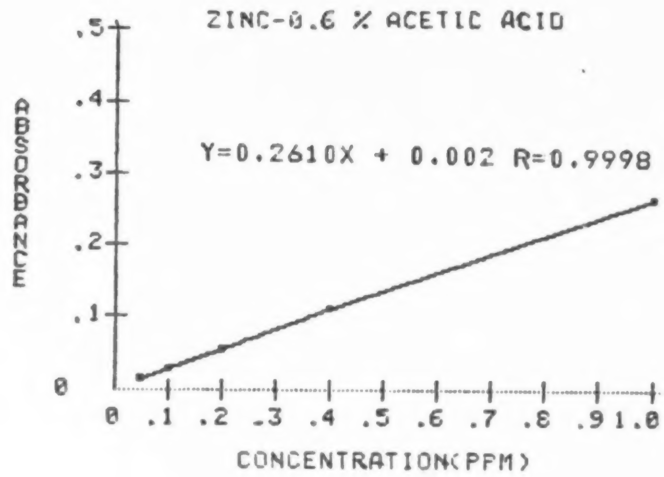

SILVER-0.5 % NITRIC ACID
Y=0.0727X + 0.001 R=0.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

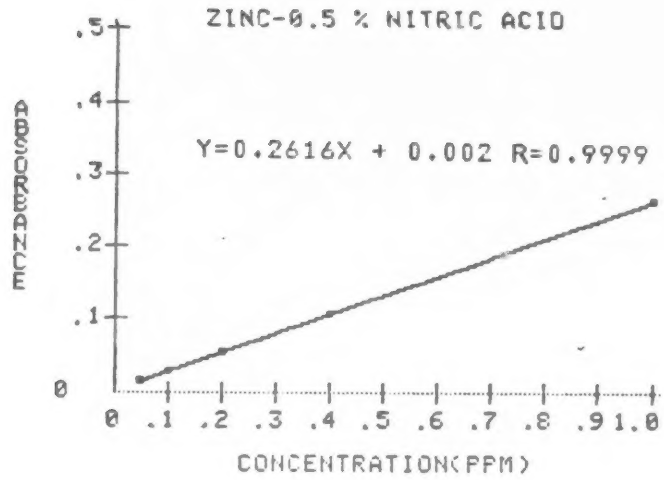

SILVER-0.6% ACETIC-0.5% NITRIC
Y=0.0738X + 0.001 R=0.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0
1
2
3
4
5
CONCENTRATION(PPM)

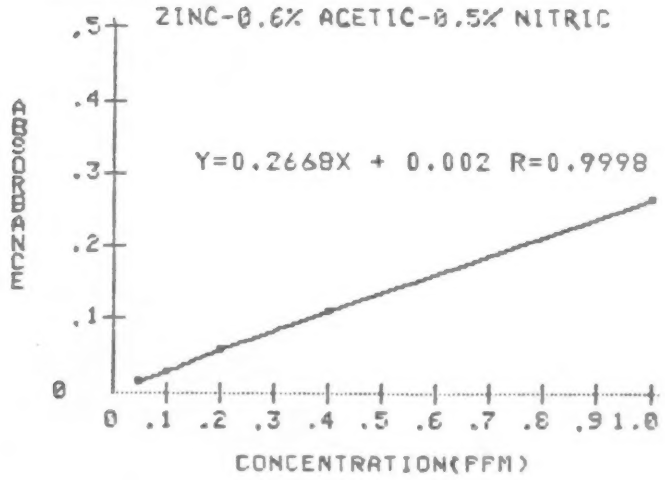

THALLIUM-0.6 % ACETIC ACID
Y=0.0101X + 0.010 R=0.9999
ABSORBANCE
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
0 5 10 15 20 25 30 35 40 45 50
CONCENTRATION(PPM)

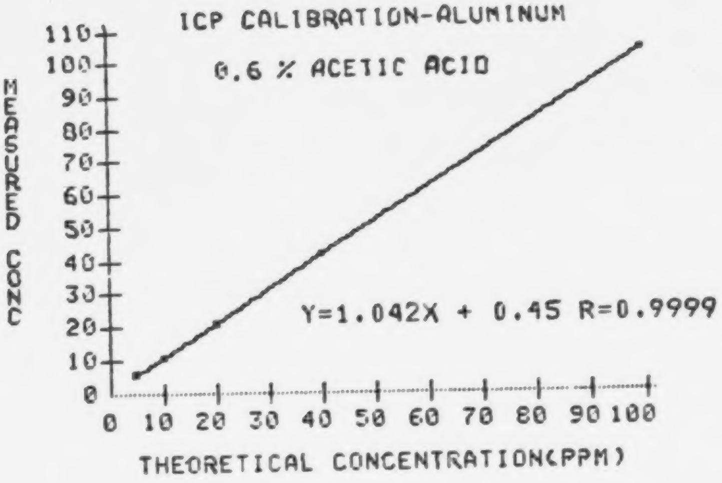

THALLIUM-0.5 % NITRIC ACID
Y=0.0099X + 0.009 R=0.9999
ABSORBANCE
0
1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
0 5 10 15 20 25 30 35 40 45 50
CONCENTRATION(PPM)

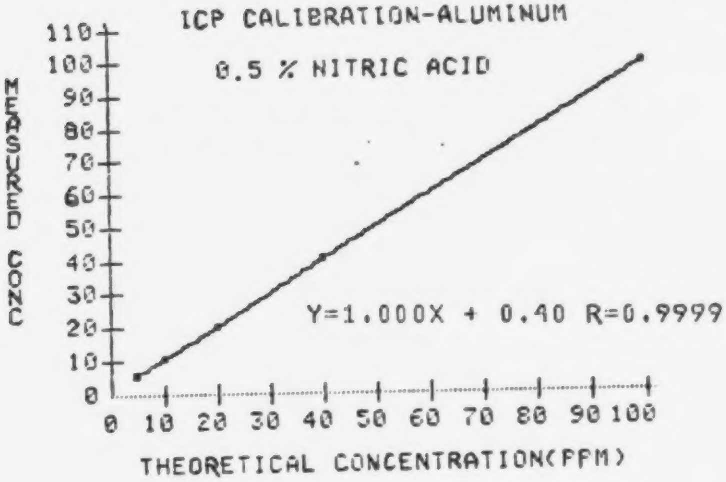

THALLIUM-0.6% ACETIC-0.5% NITRIC
Y=0.0101X + 0.002 R=0.9999
ABSORBANCE
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
0 5 10 15 20 25 30 35 40 45 50
CONCENTRATION(PPM)

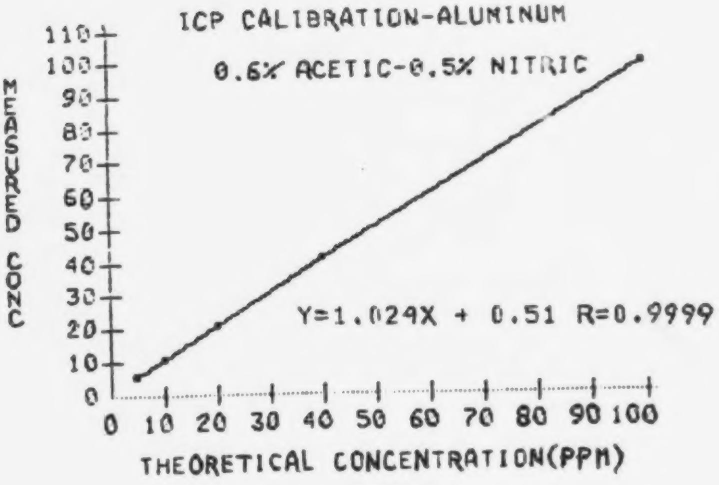

ZINC-0.6 % ACETIC ACID
Y=0.2610X + 0.002 R=0.9998
ABSORBANCE
.5
.4
.3
.2
.1
0
0 .1 .2 .3 .4 .5 .6 .7 .8 .9 1.0
CONCENTRATION(PPM)

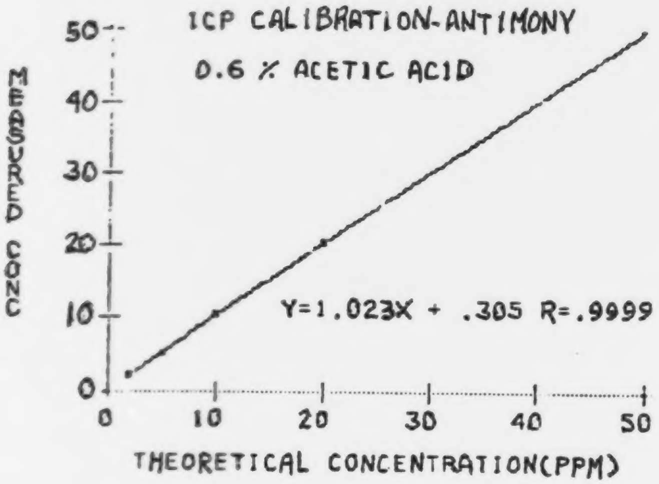

ZINC-0.5 % NITRIC ACID
Y=0.2616X + 0.002 R=0.9999
ABSORBANCE
.5
.4
.3
.2
.1
0
0 .1 .2 .3 .4 .5 .6 .7 .8 .9 1.0
CONCENTRATION(PPM)

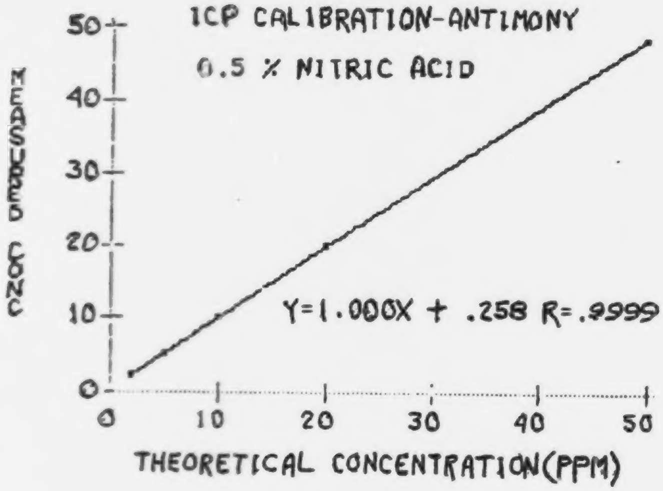

ZINC-0.6% ACETIC-0.5% NITRIC
Y=0.2668X + 0.002 R=0.9998
ABSORBANCE
.5
.4
.3
.2
.1
0
0 .1 .2 .3 .4 .5 .6 .7 .8 .9 1.0
CONCENTRATION(PPM)

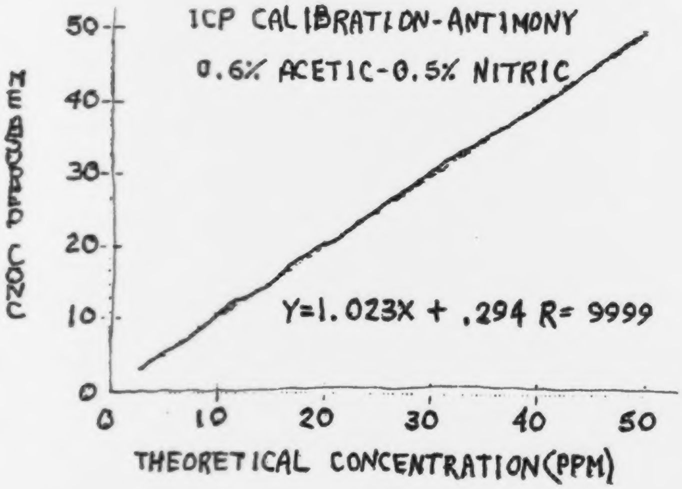

ICP CALIBRATION-ALUMINUM
0.6 % ACETIC ACID
Y=1.042X + 0.45 R=0.9999
MEASURED CONC
110
100
90
80
70
60
50
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

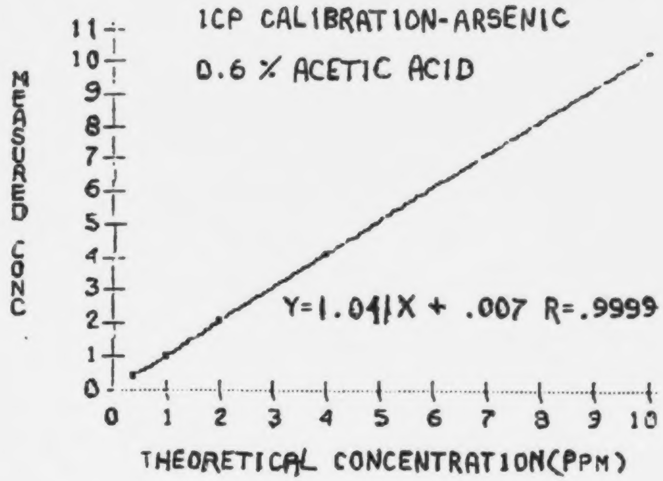

ICP CALIBRATION-ALUMINUM
0.5 % NITRIC ACID
Y=1.000X + 0.40 R=0.9999
MEASURED CONC
110
100
90
80
70
60
50
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

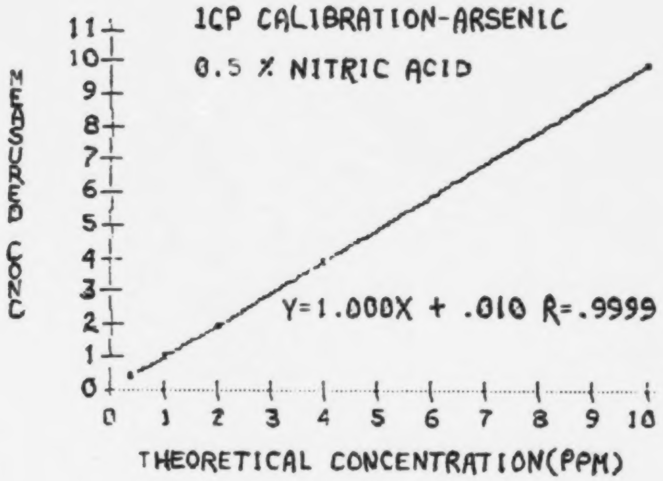

ICP CALIBRATION-ALUMINUM
0.6% ACETIC-0.5% NITRIC
Y=1.024X + 0.51 R=0.9999
MEASURED CONC
110
100
90
80
70
60
50
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

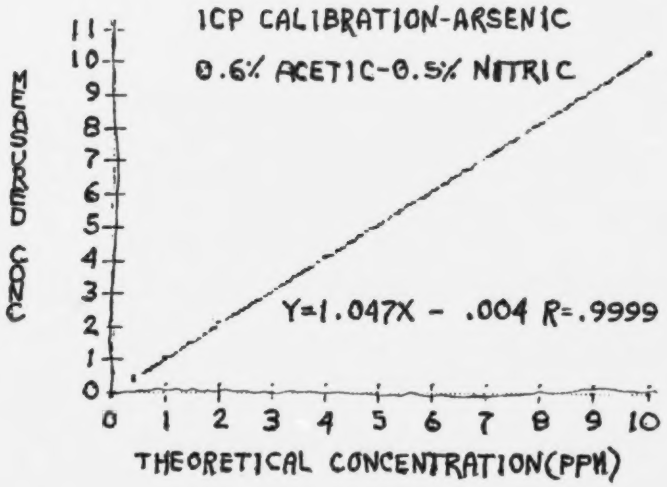

ICP CALIBRATION-ANTIMONY
0.6 % ACETIC ACID
Y=1.023X + .305 R=.9999
MEASURED CONC
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

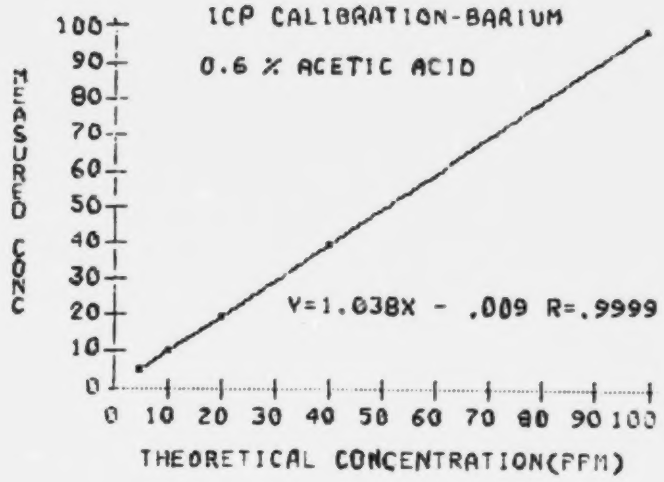

ICP CALIBRATION-ANTIMONY
0.5 % NITRIC ACID
Y=1.000X + .258 R=.9999
MEASURED CONC
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

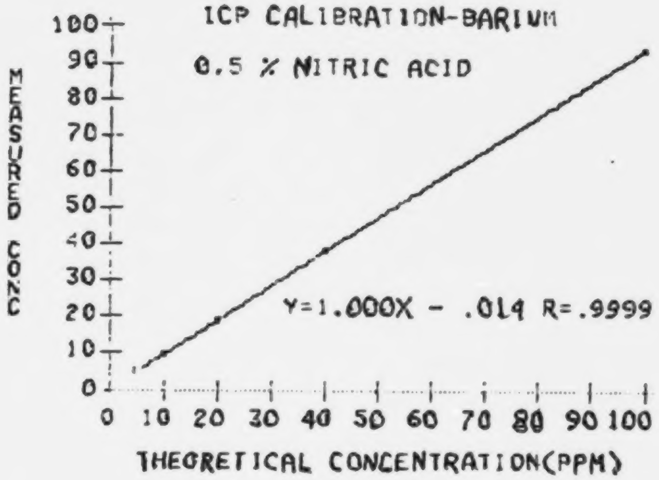

ICP CALIBRATION-ANTIMONY
0.6% ACETIC-0.5% NITRIC
Y=1.023X + .294 R= 9999
MEASURED CONC
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

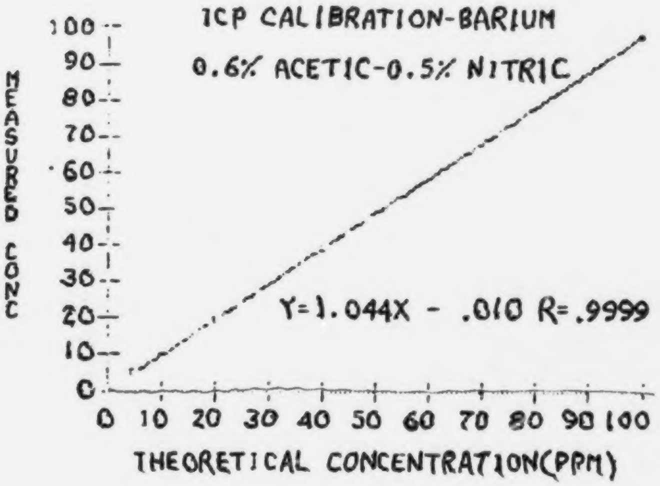

ICP CALIBRATION-ARSENIC
0.6 % ACETIC ACID
Y=1.041X + .007 R=.9999
MEASURED CONC
0
1
2
3
4
5
6
7
8
9
10
11
0
1
2
3
4
5
6
7
8
9
10
THEORETICAL CONCENTRATION(PPM)

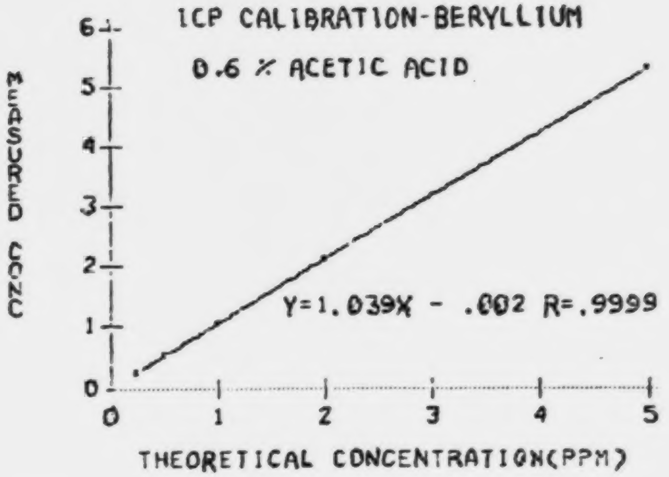

ICP CALIBRATION-ARSENIC
0.5 % NITRIC ACID
Y=1.000X + .010 R=.9999
MEASURED CONC
0
1
2
3
4
5
6
7
8
9
10
11
0
1
2
3
4
5
6
7
8
9
10
THEORETICAL CONCENTRATION(PPM)

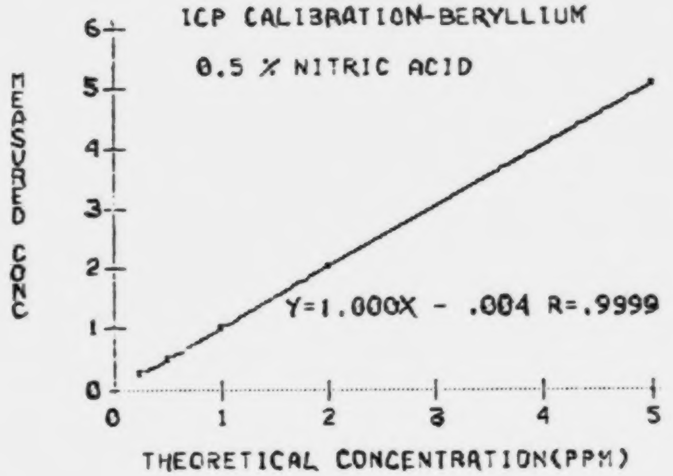

ICP CALIBRATION-ARSENIC
0.6% ACETIC-0.5% NITRIC
Y=1.047X - .004 R=.9999
MEASURED CONC
0
1
2
3
4
5
6
7
8
9
10
11
0
1
2
3
4
5
6
7
8
9
10
THEORETICAL CONCENTRATION(PPM)

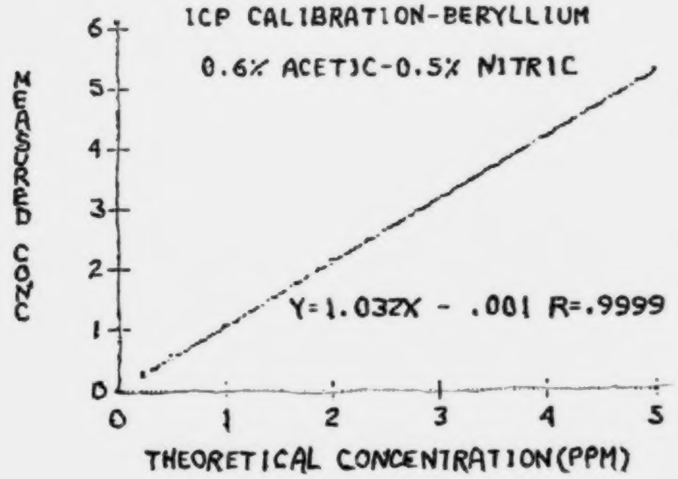

ICP CALIBRATION-BARIUM
0.6 % ACETIC ACID
Y=1.038X - .009 R=.9999
MEASURED CONC
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

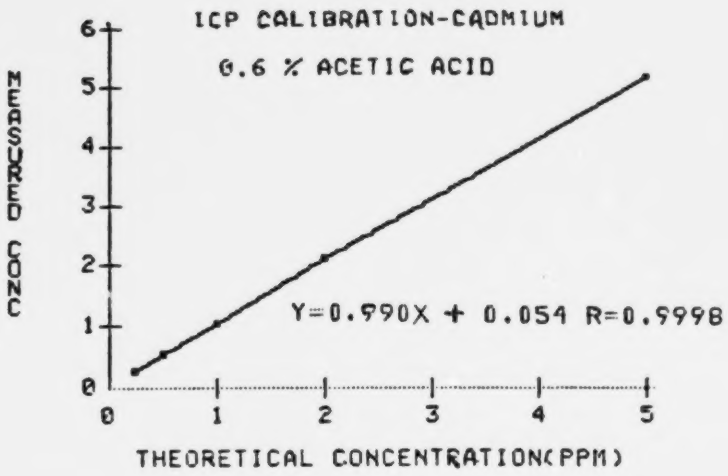

ICP CALIBRATION-BARIUM
0.5 % NITRIC ACID
Y=1.000X - .014 R=.9999
MEASURED CONC
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

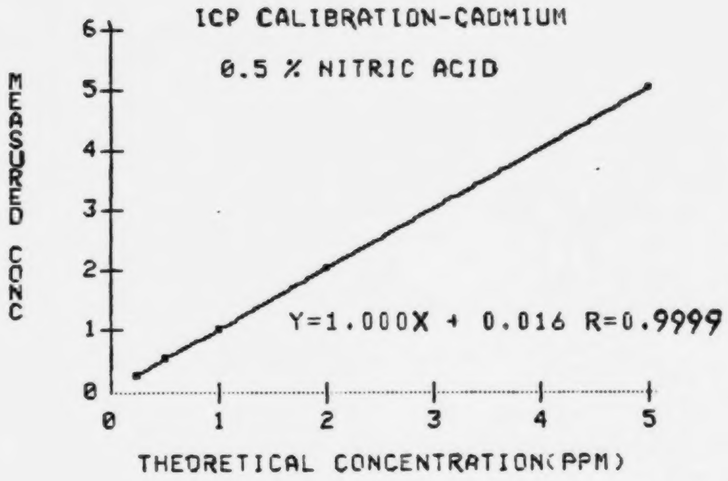

ICP CALIBRATION-BARIUM
0.6% ACETIC-0.5% NITRIC
Y=1.044X - .010 R=.9999
MEASURED CONC
0 10 20 30 40 50 60 70 80 90 100
THEORETICAL CONCENTRATION(PPM)

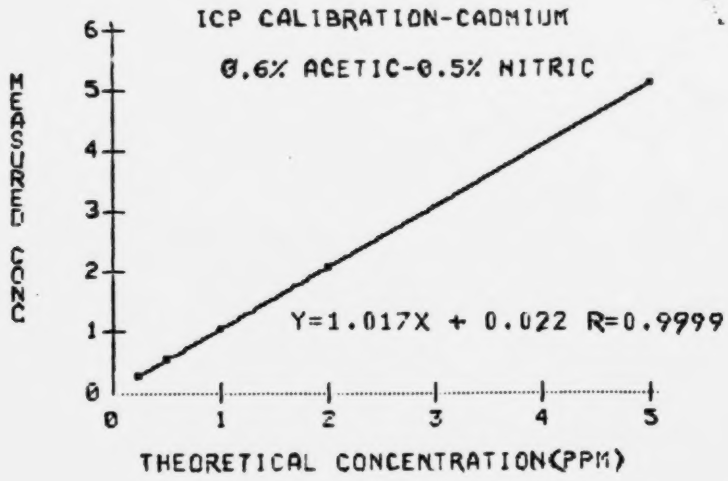

ICP CALIBRATION-BERYLLIUM
0.6 % ACETIC ACID
MEASURED CONC
Y=1.039X - .002 R=.9999
0
1
2
3
4
5
6
THEORETICAL CONCENTRATION(PPM)

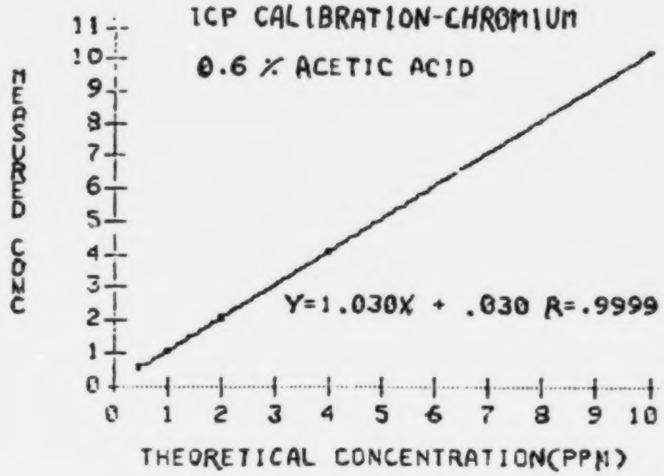

ICP CALIBRATION-BERYLLIUM
0.5 % NITRIC ACID
MEASURED CONC
Y=1.000X - .004 R=.9999
0
1
2
3
4
5
6
THEORETICAL CONCENTRATION(PPM)

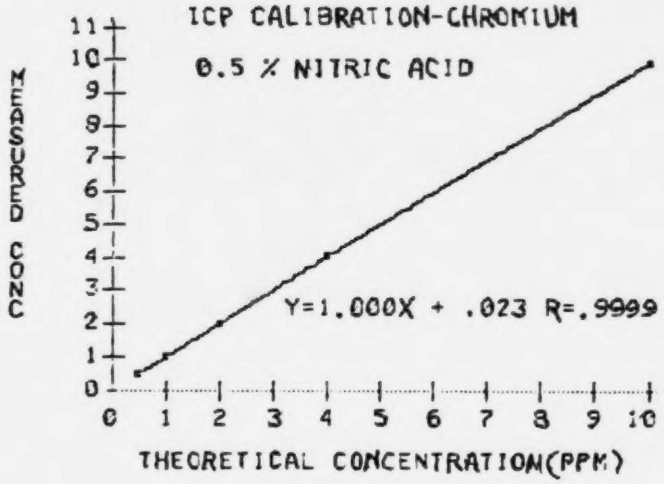

ICP CALIBRATION-BERYLLIUM
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
Y=1.032X - .001 R=.9999
0
1
2
3
4
5
6
THEORETICAL CONCENTRATION(PPM)

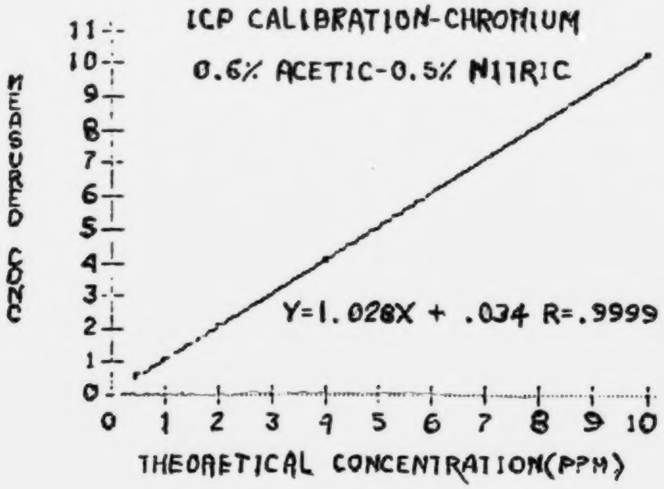

ICP CALIBRATION-CADMIUM
0.6 % ACETIC ACID
MEASURED CONC
0
1
2
3
4
5
6
Y=0.990X + 0.054 R=0.9998
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

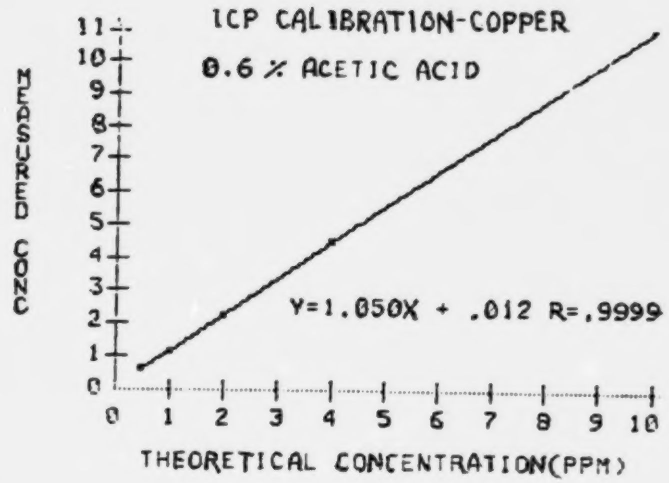

ICP CALIBRATION-CADMIUM
0.5 % NITRIC ACID
MEASURED CONC
0
1
2
3
4
5
6
Y=1.000X + 0.016 R=0.9999
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

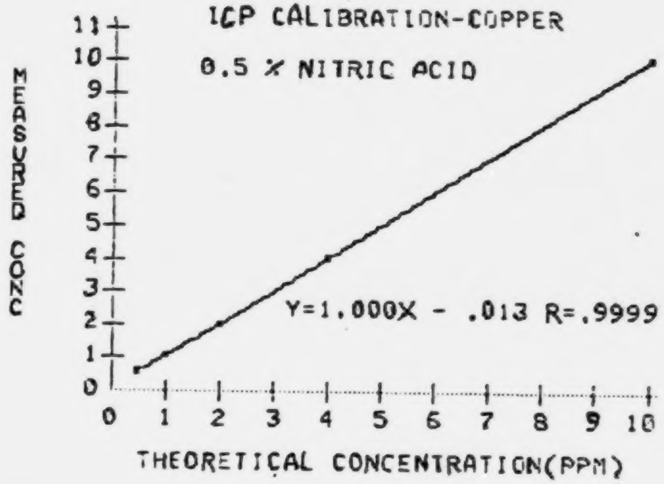

ICP CALIBRATION-CADMIUM
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
0
1
2
3
4
5
6
Y=1.017X + 0.022 R=0.9999
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

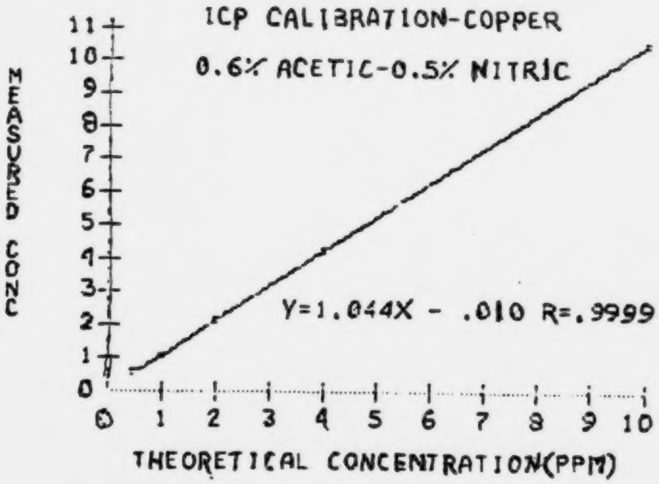

ICP CALIBRATION-CHROMIUM
0.6 % ACETIC ACID
Y=1.030X + .030 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10

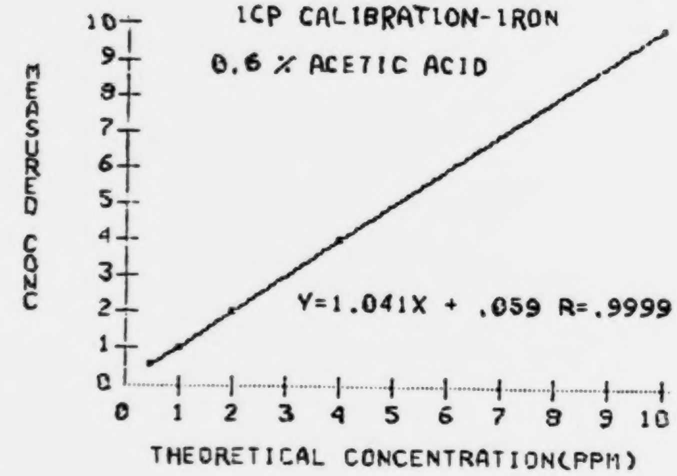

ICP CALIBRATION-CHROMIUM
0.5 % NITRIC ACID
Y=1.000X + .023 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10

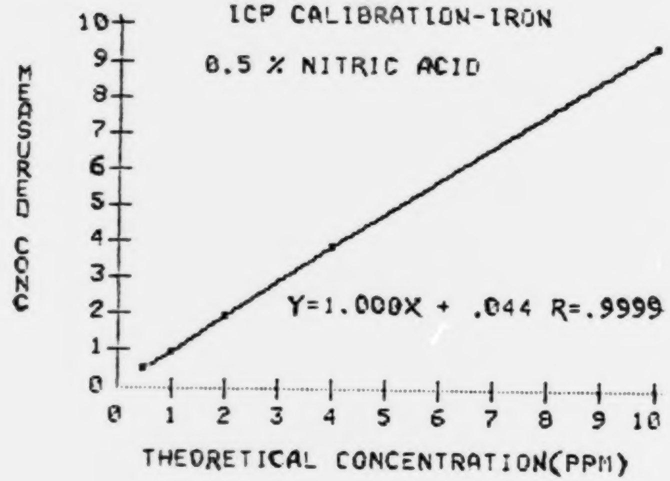

ICP CALIBRATION-CHROMIUM
0.6% ACETIC-0.5% NITRIC
Y=1.028X + .034 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10

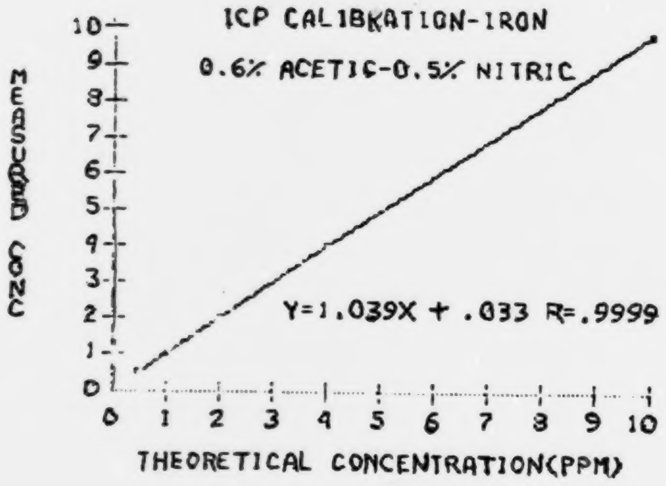

ICP CALIBRATION-COPPER
0.6 % ACETIC ACID
Y=1.050X + .012 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

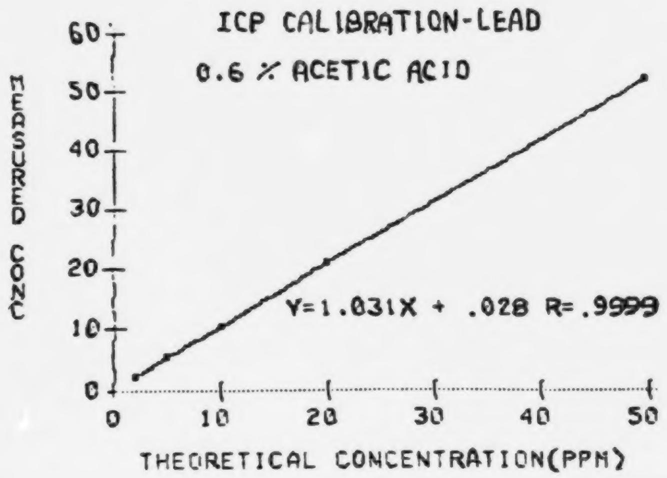

ICP CALIBRATION-COPPER
0.5 % NITRIC ACID
Y=1.000X - .013 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

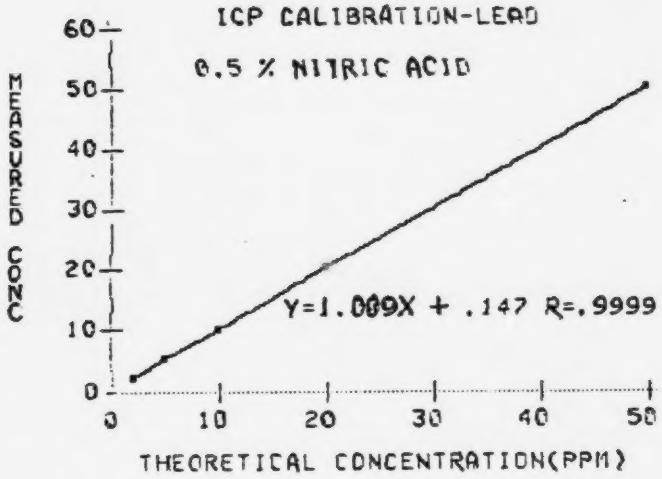

ICP CALIBRATION-COPPER
0.6% ACETIC-0.5% NITRIC
Y=1.044X - .010 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

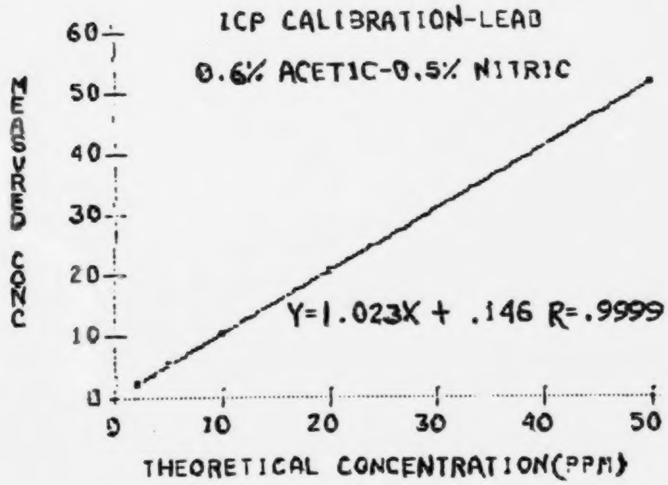

ICP CALIBRATION-IRON
0.6 % ACETIC ACID
Y=1.041X + .059 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10
0 1 2 3 4 5 6 7 8 9 10

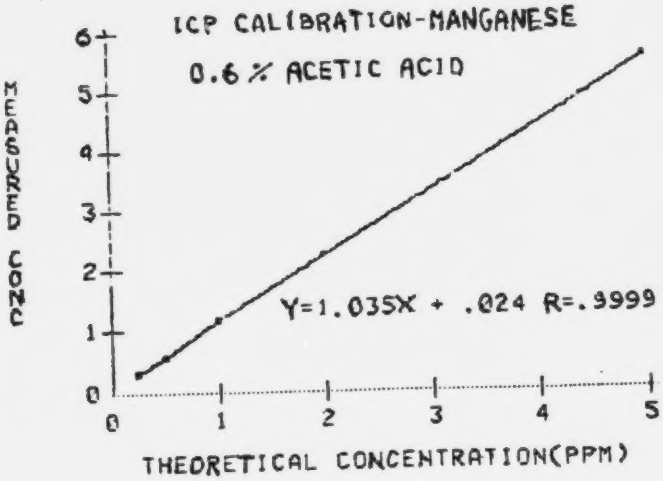

ICP CALIBRATION-IRON
0.5 % NITRIC ACID
Y=1.000X + .044 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10
0 1 2 3 4 5 6 7 8 9 10

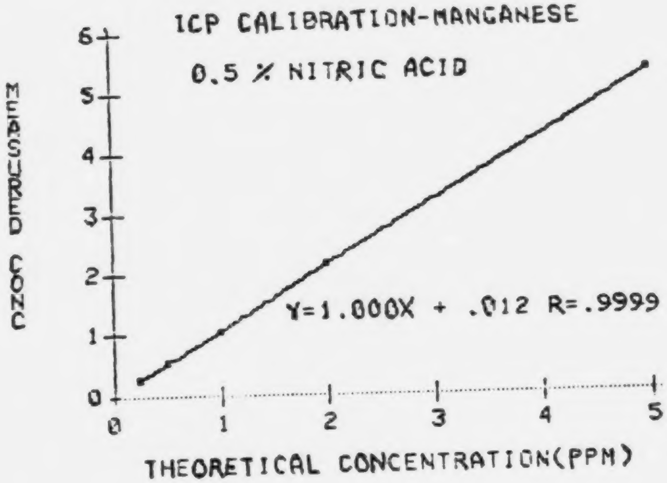

ICP CALIBRATION-IRON
0.6% ACETIC-0.5% NITRIC
Y=1.039X + .033 R=.9999
MEASURED CONC
THEORETICAL CONCENTRATION(PPM)
0 1 2 3 4 5 6 7 8 9 10
0 1 2 3 4 5 6 7 8 9 10

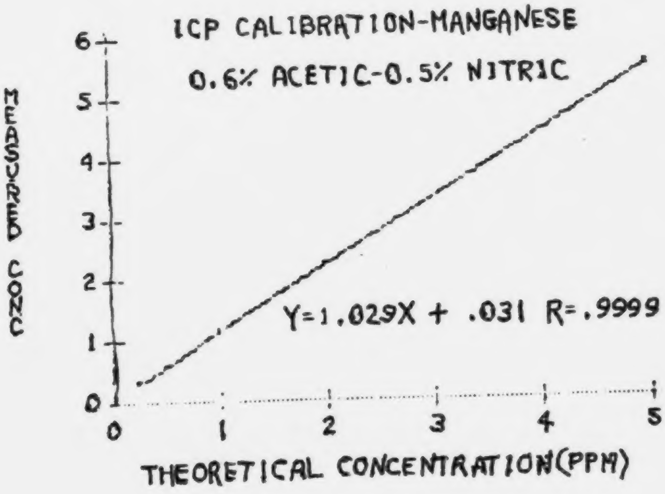

ICP CALIBRATION-LEAD
0.6 % ACETIC ACID
Y=1.031X + .028 R=.9999
MEASURED CONC
60
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

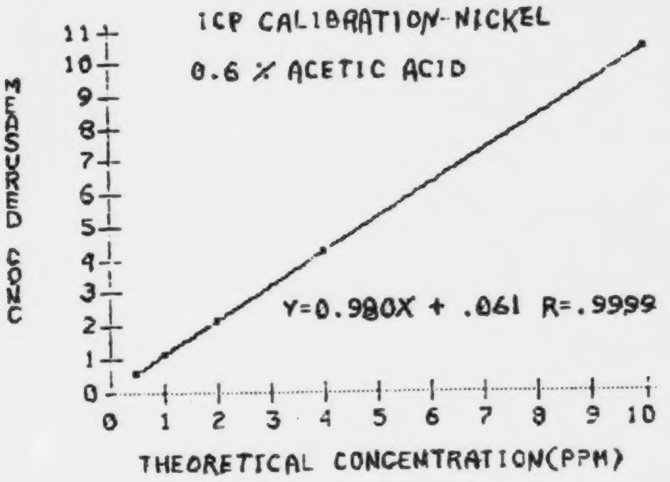

ICP CALIBRATION-LEAD
0.5 % NITRIC ACID
Y=1.009X + .147 R=.9999
MEASURED CONC
60
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

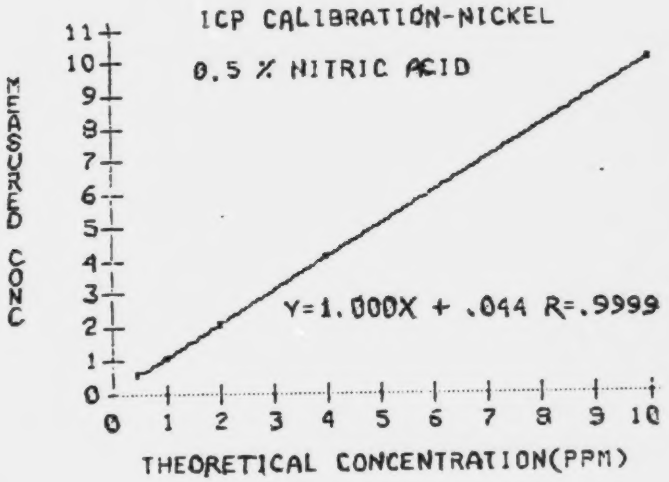

ICP CALIBRATION-LEAD
0.6% ACETIC-0.5% NITRIC
Y=1.023X + .146 R=.9999
MEASURED CONC
60
50
40
30
20
10
0
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)

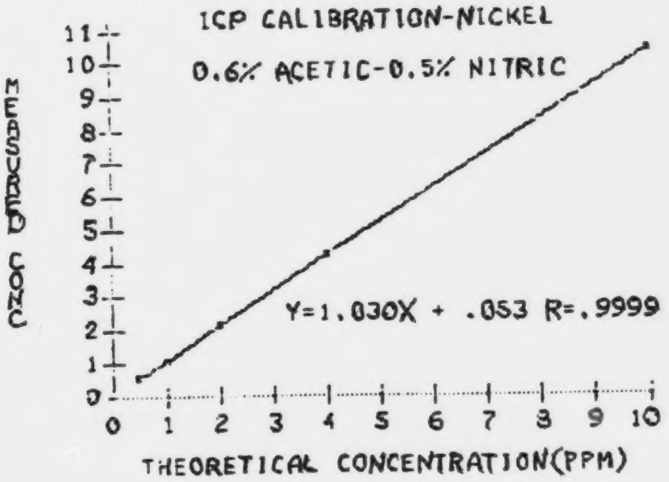

ICP CALIBRATION-MANGANESE
0.6 % ACETIC ACID
MEASURED CONC
0
1
2
3
4
5
6
Y=1.035X + .024 R=.9999
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

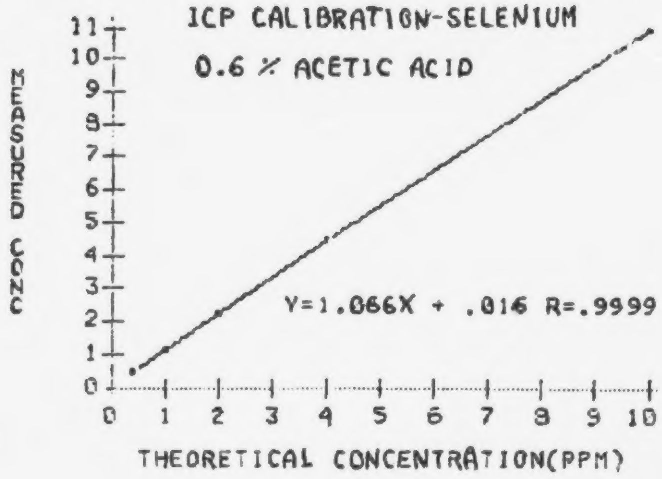

ICP CALIBRATION-MANGANESE
0.5 % NITRIC ACID
MEASURED CONC
0
1
2
3
4
5
6
Y=1.000X + .012 R=.9999
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

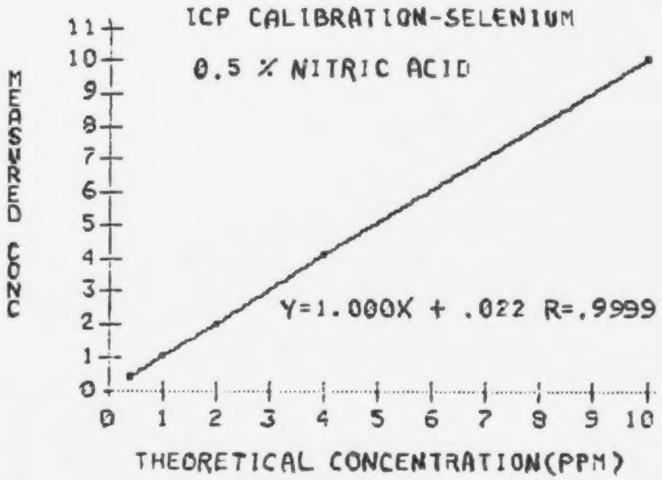

ICP CALIBRATION-MANGANESE
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
0
1
2
3
4
5
6
Y=1.029X + .031 R=.9999
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

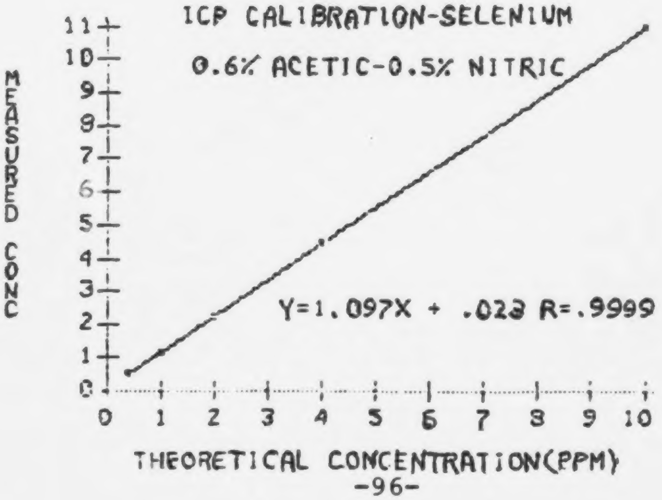

ICP CALIBRATION-NICKEL
0.6 % ACETIC ACID
MEASURED CONC
Y=0.980X + .061 R=.9999
0
1
2
3
4
5
6
7
8
9
10
11
THEORETICAL CONCENTRATION(PPM)

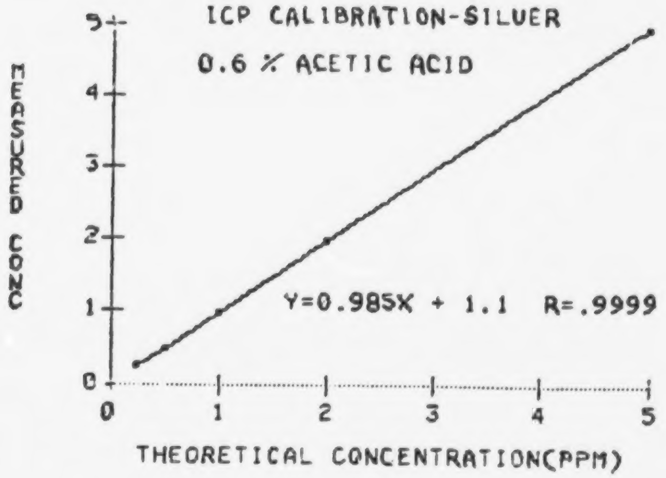

ICP CALIBRATION-NICKEL
0.5 % NITRIC ACID
MEASURED CONC
Y=1.000X + .044 R=.9999
0
1
2
3
4
5
6
7
8
9
10
11
THEORETICAL CONCENTRATION(PPM)

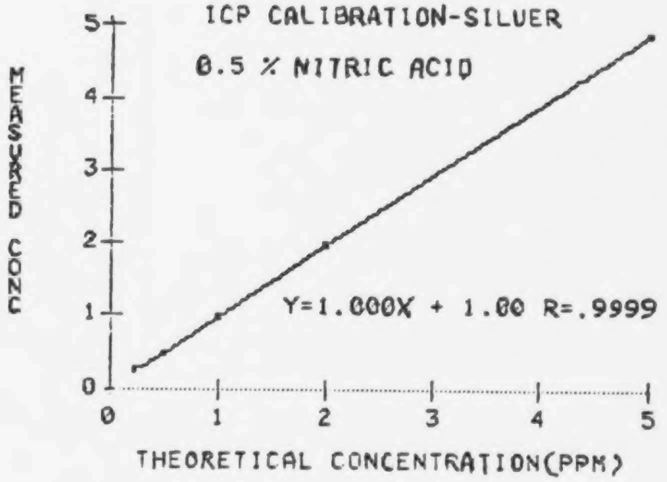

ICP CALIBRATION-NICKEL
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
Y=1.030X + .053 R=.9999
0
1
2
3
4
5
6
7
8
9
10
11
THEORETICAL CONCENTRATION(PPM)

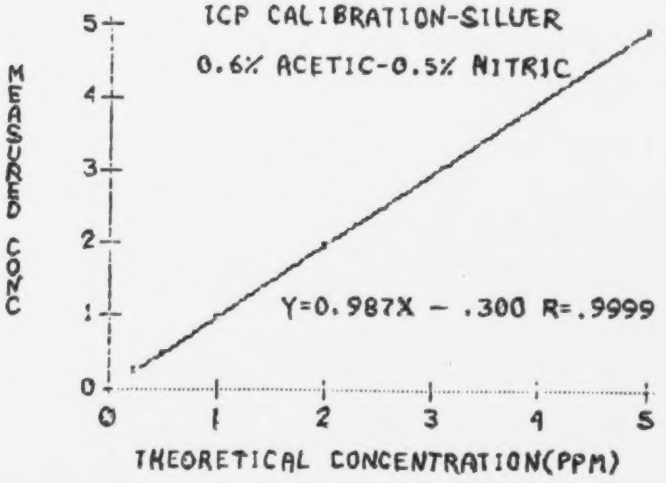

ICP CALIBRATION-SELENIUM
0.6 % ACETIC ACID
Y=1.066X + .016 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

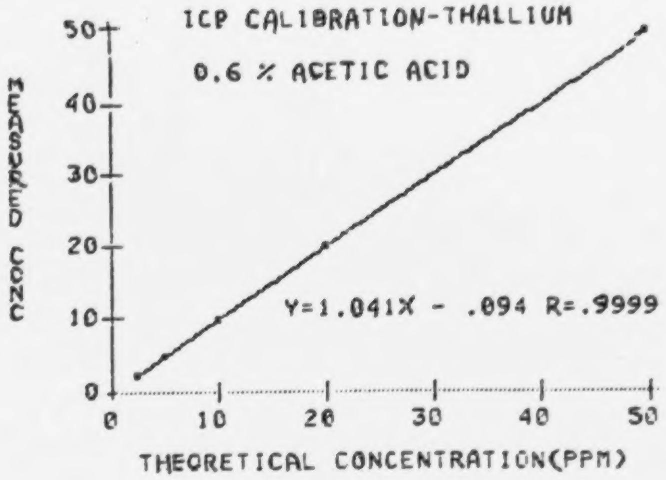

ICP CALIBRATION-SELENIUM
0.5 % NITRIC ACID
Y=1.000X + .022 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

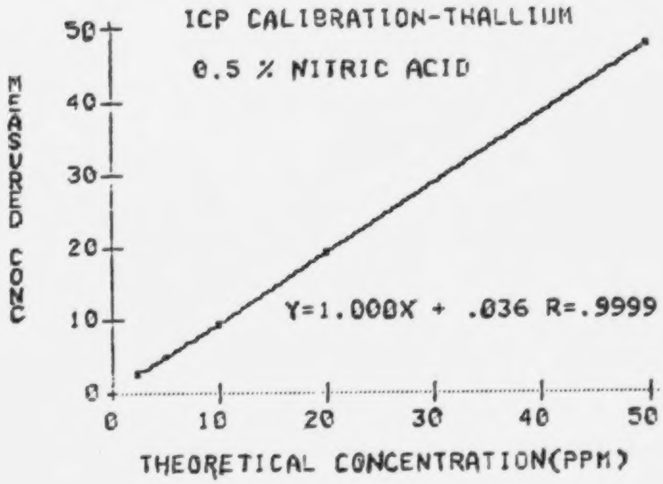

ICP CALIBRATION-SELENIUM
0.6% ACETIC-0.5% NITRIC
Y=1.097X + .023 R=.9999
MEASURED CONC
0 1 2 3 4 5 6 7 8 9 10 11
0 1 2 3 4 5 6 7 8 9 10
THEORETICAL CONCENTRATION(PPM)

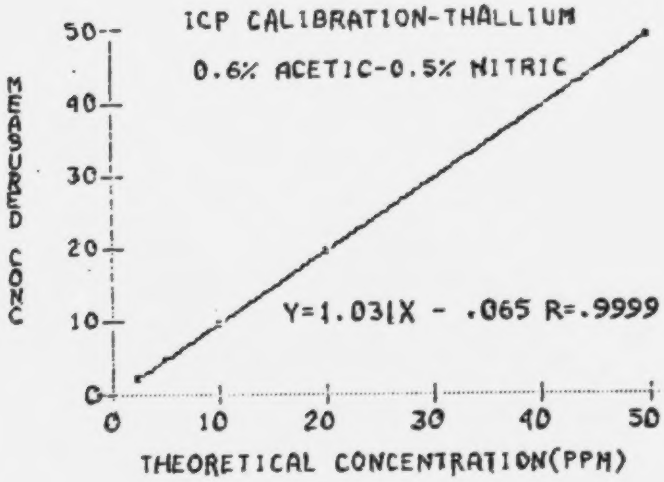

ICP CALIBRATION-SILVER
0.6 % ACETIC ACID
MEASURED CONC
Y=0.985X + 1.1 R=.9999
0
1
2
3
4
5
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

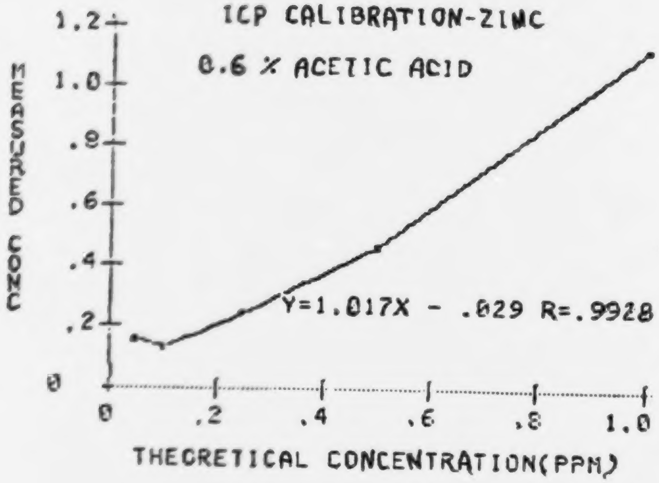

ICP CALIBRATION-SILVER
0.5 % NITRIC ACID
MEASURED CONC
Y=1.000X + 1.00 R=.9999
0
1
2
3
4
5
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

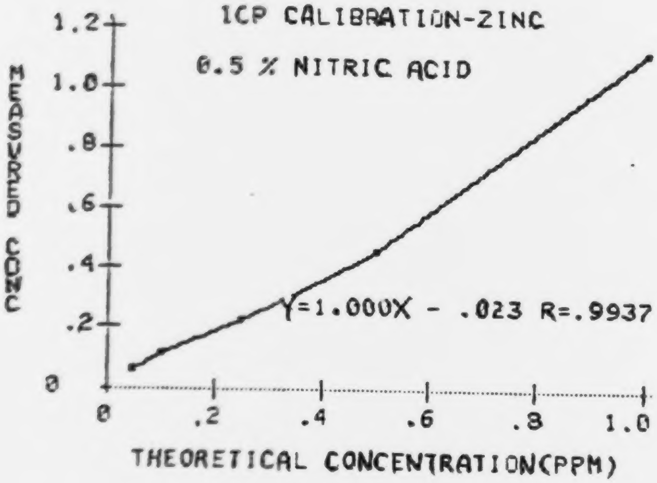

ICP CALIBRATION-SILVER
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
Y=0.987X - .300 R=.9999
0
1
2
3
4
5
0
1
2
3
4
5
THEORETICAL CONCENTRATION(PPM)

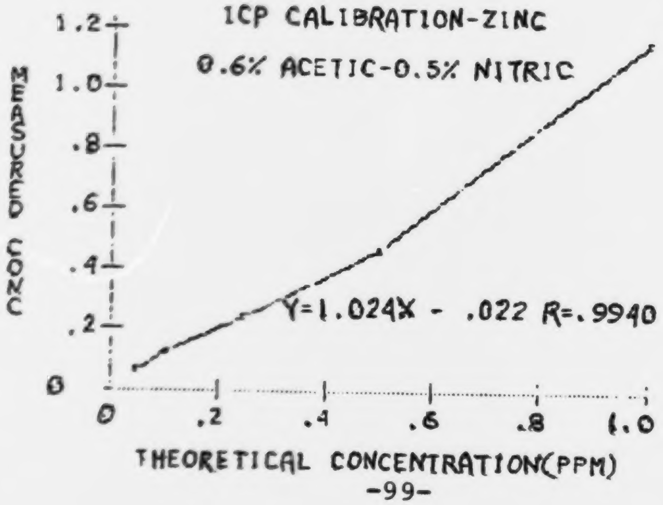

ICP CALIBRATION-THALLIUM
0.6 % ACETIC ACID
Y=1.041X - .094 R=.9999
MEASURED CONC
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)


ICP CALIBRATION-THALLIUM
0.5 % NITRIC ACID
Y=1.000X + .036 R=.9999
MEASURED CONC
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)


ICP CALIBRATION-THALLIUM
0.6% ACETIC-0.5% NITRIC
Y=1.031X - .065 R=.9999
MEASURED CONC
0
10
20
30
40
50
THEORETICAL CONCENTRATION(PPM)


ICP CALIBRATION-ZINC
0.6 % ACETIC ACID
MEASURED CONC
1.2
1.0
.8
.6
.4
.2
0
Y=1.017X - .029 R=.9928
0
.2
.4
.6
.8
1.0
THEORETICAL CONCENTRATION(PPM)


ICP CALIBRATION-ZINC
0.5 % NITRIC ACID
MEASURED CONC
1.2
1.0
.8
.6
.4
.2
0
Y=1.000X - .023 R=.9937
0
.2
.4
.6
.8
1.0
THEORETICAL CONCENTRATION(PPM)


ICP CALIBRATION-ZINC
0.6% ACETIC-0.5% NITRIC
MEASURED CONC
1.2
1.0
.8
.6
.4
.2
0
Y=1.024X - .022 R=.9940
0
.2
.4
.6
.8
1.0
THEORETICAL CONCENTRATION(PPM)

APPENDIX B

RAW DATA FROM PHASE 1 ANALYSES OF RIVER SEDIMENT

This appendix summarizes Phase 1 analyses performed on samples of river sediment. The Phase 1 procedures consisted of the following general analytical schemes:

Scheme 1 - EP test with leachate digestion

Scheme 2 - EP test without leachate digestion

Scheme 3 - digestion according to EPA (1979a) with sample stored at room temperature

Scheme 4 - digestion according to EPA (1979a) with sample preserved by addition of nitric acid (HNO_3) to pH <2

Results of determinations from Schemes 1 and 2 were used to evaluate the requirement to digest leachates resulting from the EP test. Results of determinations from Schemes 3 and 4 were used to evaluate sample storage requirements.

Raw data from the Phase 1 analyses of river sediment are presented in Table B-1. The mean and standard deviation for elemental determinations by ICP and AAS are summarized for each analytical scheme in Table B-2. Recoveries of metal spikes from sample digestates and leachates (after storage for two weeks) are presented in Table B-3.

TABLE B-1. RESULTS OF PHASE I ANALYSES OF RIVER SEDIMENT.

		Concentration[a]											
		Scheme 1[b] (ERCO ID TM-81-1230)			Scheme 2[c] (ERCO ID TM-81-1232)			Scheme 3[d] (ERCO ID TM-81-1233)			Scheme 4[e] (ERCO ID TM-81-1231)		
Element	Method	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3
Al	ICP	0.088	0.268	0.109	0.338	0.195	0.108	2,970	2,670	2,550	2,600	2,860	2,490
	AAS	<0.040	0.242	0.080	<0.250	<0.250	<0.250	3,000	2,990	3,170	2,890	3,170	2,850
Sb	ICP	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	0.73	0.86	1.2	0.89	1.4	1.0
	AAS	<0.080	<0.080	<0.080	<0.500	<0.500	<0.500	<10	<10	<10	<10	<10	<10
As	ICP	<0.016	<0.016	<0.016	<0.080	<0.080	<0.080	5.0	5.4	5.0	4.4	4.5	5.2
	AAS	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	1.9	1.5	1.5	0.97	0.97	1.0
Ba	ICP	0.097	0.176	0.220	0.350	0.282	0.200	5.9	15	22	19	21	20
	AAS	0.117	0.139	0.197	<0.25	<0.25	<0.25	19	20	21	16	23	20
Be	ICP	0.0003	0.0007	0.0002	0.0001	0.0005	0.0004	0.25	0.24	0.23	0.23	0.27	0.24
	AAS	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	0.18	0.17	0.18	0.18	0.23	0.16
Cd	ICP	0.006	0.008	0.005	0.009	0.006	0.005	0.34	0.33	0.34	0.33	0.33	0.38
	AAS	0.003	0.009	0.005	<0.010	<0.010	<0.010	0.42	0.37	0.47	0.32	0.39	0.39
Cr	ICP	0.003	0.003	0.003	<0.015	<0.015	<0.015	19	18	18	17	19	19
	AAS	<0.030	<0.030	<0.030	<0.18	<0.18	<0.18	16	19	19	18	16	18
Cu	ICP	0.004	0.023	0.014	<0.015	<0.015	<0.015	16	16	15	16	16	19
	AAS	<0.020	<0.020	<0.020	<0.100	<0.100	<0.100	18	19	19	21	18	20
Fe	ICP	0.105	0.049	0.055	0.127	0.057	0.078	4,190	4,400	4,190	4,250	4,410	3,970
	AAS	0.120	0.038	0.053	<0.100	0.135	0.135	4,700	5,200	4,800	3,500	3,700	4,300
Pb	ICP	0.024	0.403	0.128	0.389	0.243	0.063	380	370	400	340	460	415
	AAS	<0.040	0.350	0.100	<0.200	<0.200	<0.200	350	350	380	320	440	390
Mn	ICP	1.06	2.65	2.12	2.65	2.38	2.09	96	98	94	94	97	91
	AAS	1.10	2.67	2.36	2.47	2.22	2.78	100	100	100	97	100	99
Hg (ug/L)	ICP	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
	AAS	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.064	0.089	0.076	0.068	0.069	0.067
Ni	ICP	0.010	0.029	0.020	0.027	0.026	0.019	4.3	4.1	3.9	4.2	4.1	4.2
	AAS	<0.010	0.023	0.023	<0.050	<0.050	<0.050	4.6	4.9	4.6	4.3	4.5	4.5
Se	ICP	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	<5	<5	<5	<5	<5	<5
	AAS	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
Ag	ICP	<0.001	<0.001	<0.001	<0.005	<0.005	<0.005	0.19	0.24	0.22	0.23	0.16	0.19
	AAS	<0.005	<0.005	<0.005	<0.025	<0.025	<0.025	0.30	0.29	0.29	0.29	0.24	0.24
Tl	ICP	<0.030	<0.030	<0.015	<0.015	<0.015	<0.030	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
	AAS	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Zn	ICP	0.220	1.20	0.780	1.06	0.952	0.681	57	57	59	55	59	59
	AAS	0.200	0.960	0.720	1.03	0.936	0.669	51	50	52	48	50	50

[a] Unless otherwise noted, concentrations are mg/L for Schemes 1 and 2; ug/g for Schemes 3 and 4.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA^2. Sample stored at room temperature.

[e] Digestion according to EPA_2. Sample preserved with HNO_3 to pH <2.

TABLE B-2. SUMMARY OF PHASE 1 ANALYSES OF RIVER SEDIMENT CONCENTRATION

Element	Method	Concentration[a]			
		Scheme 1[b] mg/L	Scheme 2[c] mg/L	Scheme 3[d] ug/g	Scheme 4[e] ug/g
Al	ICP	0.155 ± 0.099	0.214 ± 0.116	2,730 ± 216	2,650 ± 190
	AAS	0.121 ± 0.107	<0.250[f]	3,053 ± 101	2,746 ± 222
Sb	ICP	<0.010[f]	<0.050[f]	0.94 ± 0.25	1.1 ± 0.28
	AAS	<0.080[f]	<0.500[f]	<10[f]	<10[f]
As	ICP	<0.016[f]	<0.080[f]	5.1 ± 0.23	4.7 ± 0.44
	AAS	<0.002[f]	<0.010[f]	1.6 ± 0.23	0.98 ± 0.02
Ba	ICP	0.164 ± 0.062	0.277 ± 0.075	14 ± 8.1	20 ± 1.0
	AAS	0.151 ± 0.041	<0.250[f]	20 ± 1.0	20 ± 3.5
Be	ICP	0.0004±0.0003	0.0003±0.0002	0.24 ± 0.01	0.25 ± 0.02
	AAS	<0.002[f]	<0.010[f]	0.18 ± 0.01	0.19 ± 0.04
Cd	ICP	0.006 ± 0.002	0.007 ± 0.002	0.34 ± 0.01	0.35 ± 0.03
	AAS	0.006 ± 0.003	<0.010[f]	0.42 ± 0.05	0.37 ± 0.04
Cr	ICP	0.003 ± 0.0	<0.015[f]	18 ± 0.58	18 ± 1.2
	AAS	<0.03[f]	<0.180[f]	18 ± 1.7	17 ± 1.2
Cu	ICP	0.014 ± 0.010	<0.015[f]	16 ± 0.58	17 ± 1.7
	AAS	<0.02[f]	<0.100[f]	19 ± 0.58	20 ± 1.5
Fe	ICP	0.070 ± 0.031	0.088 ± 0.036	4,260 ± 121	4,210 ± 223
	AAS	0.070 ± 0.044	0.123 ± 0.020	4,900 ± 265	3,833 ± 416
Pb	ICP	0.185 ± 0.196	0.232 ± 0.163	383 ± 15	405 ± 61
	AAS	0.160 ± 0.160	<0.200[f]	360 ± 17	383 ± 60
Mn	ICP	1.94 ± 0.81	2.37 ± 0.28	96 ± 1.9	94 ± 3.1
	AAS	2.04 ± 0.83	2.49 ± 0.28	100 ± 0.0	99 ± 1.5
Hg	ICP	<10	<10	<10	<10
	AAS	<0.01 ug/1[f]	<0.01 ug/1[f]	0.076 ± 0.013	0.068 ± 0.001
Ni	ICP	0.020 ± 0.010	0.024 ± 0.004	4.1 ± 0.16	4.2 ± 0.02
	AAS	0.019 ± 0.008	<0.050[f]	4.7 ± 0.18	4.5 ± 0.15
Se	ICP	<0.010[f]	<0.050[f]	<5[f]	<5[f]
	AAS	<0.002[f]	<0.010[f]	<0.10[f]	<0.10[f]
Ag	ICP	<0.001[f]	<0.005[f]	0.22 ± 0.02	0.19 ± 0.03
	AAS	<0.005[f]	<0.025[f]	0.29 ± 0.01	0.25 ± 0.03
Tl	ICP	<0.03[f]	<0.15[f]	<2.0[f]	<2.0[f]
	AAS	<0.04[f]	<0.20[f]	<2.0[f]	<2.0[f]
Zn	ICP	0.730 ± 0.487	0.898 ± 0.196	58 ± 1.2	58 ± 2.3
	AAS	0.626 ± 0.389	0.879 ± 0.188	51 ± 1.0	49 ± 1.2

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[a] Mean and standard deviation; unless otherwise noted units are mg/L in the extract for Schemes 1 and 2; ug/g for Scheme 3 and 4.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

[f] Mean was not calculated since one or more analyses were below the limit of detection.

TABLE B-3. SPIKE RECOVERIES FOR PHASE 1 ANALYSES OF RIVER SEDIMENT[a]

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 1, Replicate 1																	
Solution[b]	<0.025	<0.25	<0.010	0.324	<0.01	<0.1	<0.2	0.110	<0.1	6.47	<0.1	<0.25	<0.2	<0.010	<0.20	2.64	0.026
Actual spike[c]	1.29	44	0.073	24	2.59	2.43	5.29	4.90	5.21	2.79	4.70	25	23	0.056	5.40	0.38	0.23
Theor. spike[d]	1.25	46	0.050	24	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	103%	96%	146%	100%	103%	97%	106%	98%	104%	111%	95%	100%	92%	112%	108%	76%	92%
Scheme 1, Replicate 2																	
Solution	<0.025	<0.25	<0.010	0.468	<0.01	0.05	<0.2	0.110	<0.1	15.70	0.130	2.01	<0.2	<0.010	<0.20	2.40	0.021
Actual spike	1.06	44	0.075	24	2.59	2.43	5.41	4.97	5.21	2.52	4.88	25	23	0.058	5.40	0.38	0.25
Theor. spike	1.25	46	0.050	24	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	85%	96%	149%	100%	103%	97%	108%	99%	104%	101%	98%	100%	92%	116%	108%	76%	100%
Scheme 1, Replicate 3																	
Solution	<0.025	<0.25	<0.010	0.540	<0.01	0.029	<0.2	0.036	<0.1	13.93	<0.1	0.61	<0.2	<0.010	<0.20	1.21	0.020
Actual spike	1.35	45	0.064	23	2.59	2.43	5.25	4.93	5.21	2.61	4.83	25	23	0.056	5.40	0.51	0.25
Theor. spike	1.25	46	0.050	24	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	107%	98%	128%	96%	103%	97%	105%	98%	104%	105%	97%	100%	92%	112%	108%	102%	100%

[a] All concentrations are in ppm unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

Table B-3. Continued

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 2, Replicate 1																	
Solution[b]	<0.025	<0.5	<0.010	<0.5	<0.01	<0.01	<0.2	<0.025	<0.1	2.45	0.053	<0.25	<0.20	<0.010	<0.20	1.03	0.022
Actual spike[c]	1.37	44	0.071	24	1.60	2.49	5.49	5.02	5.13	2.52	4.77	26	23	0.053	5.50	0.34	0.24
Theor. spike[d]	1.25	46	0.050	24	1.66	2.5	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	109%	96%	142%	100%	96%	99%	110%	100%	102%	100%	95%	104%	92%	106%	110%	68%	98%
Scheme 2, Replicate 2																	
Solution	<0.025	<0.5	<0.010	<0.5	<0.01	<0.01	<0.2	<0.25	<0.1	2.20	<0.05	<0.25	<0.20	<0.010	<0.20	0.94	0.02
Actual spike	1.36	44	0.075	24	1.60	2.49	5.33	5.09	5.17	2.55	4.83	26	24	0.056	5.40	0.35	0.25
Theor. spike	1.25	46	0.050	24	1.66	2.5	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	108%	96%	150%	100%	96%	99%	106%	102%	103%	102%	97%	104%	96%	112%	108%	69%	100%
Scheme 2, Replicate 3																	
Solution	<0.025	<0.5	<0.010	<0.5	<0.01	<0.01	<0.2	<0.025	<0.1	2.78	<0.05	<0.25	<0.20	<0.010	<0.20	0.68	0.020
Actual spike	1.33	44	0.060	24	1.63	2.49	5.45	4.96	5.17	2.16	4.83	25	23	0.053	5.40	0.34	0.25
Theor. spike	1.25	46	0.050	24	1.66	2.5	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	106%	96%	120%	100%	98%	99%	109%	99%	103%	86%	97%	100%	92%	106%	108%	68%	100%

[a] All concentrations are in mg/L unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

Table B-3. Continued

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 3, Replicate 1																	
Solution[b]	0.047	3.0	0.024	1.03	0.023	0.060	2.31	2.64	--[e]	0.12	0.54	40.5	<0.20	<0.020	<0.20	0.06	0.067
Actual spike[c]	1.31	23	0.054	25	1.58	1.69	3.51	6.39	--[e]	1.32	3.19	15.5	14.8	0.053	5.40	0.23	0.33
Theor. spike[d]	1.25	25	0.050	24	1.66	1.66	3.33	6.66	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	104%	91%	108%	104%	95%	102%	105%	96%	--[e]	80%	96%	94%	90%	106%	108%	77%	132%
Scheme 3, Replicate 2																	
Solution	0.039	3.7	0.019	1.17	0.029	0.050	2.03	2.22	--[e]	0.13	0.58	56.8	<0.020	<0.020	<0.20	0.06	0.069
Actual spike	1.34	23	0.059	26	1.57	1.69	3.69	6.53	--[e]	1.35	3.65	15.5	15.8	0.050	5.80	0.24	0.19
Theor. spike	1.25	25	0.050	24	1.66	1.66	3.33	6.66	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	107%	93%	117%	108%	95%	102%	110%	98%	--[e]	81%	110%	93%	95%	100%	116%	73%	76%
Scheme 3, Replicate 3																	
Solution	0.025	3.6	0.016	0.75	0.017	0.040	1.94	2.07	--[e]	0.10	0.46	40.5	<0.20	<0.020	<0.20	0.06	0.067
Actual spike	1.31	24	0.052	24	1.59	1.70	3.62	6.58	--[e]	1.35	3.46	16.1	15.8	0.046	5.80	0.23	0.14
Theor. spike	1.25	25	0.050	24	1.66	1.66	3.33	6.66	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	105%	96%	103%	100%	96%	102%	109%	99%	--[e]	81%	104%	97%	95%	92%	116%	79%	56%

[a] All concentrations are in mg/L unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

[e] Insufficient sample to complete analysis.

Table B-3. Continued

Sample Identification	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
	Element																
Scheme 4, Replicate 1																	
Solution[b]	0.03	3.23	0.011	1.44	0.020	0.046	1.78	0.02	--[e]	0.11	0.51	29.9	<0.20	<0.020	<0.20	5.68	0.064
Actual spike[c]	1.33	23	0.061	25	1.75	1.60	4.90	4.93	--[e]	1.31	3.39	17.8	14.8	0.052	5.67	0.04	0.24
Theor. spike[d]	1.25	25	0.050	25	1.66	1.66	3.33	5.00	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	106%	92%	122%	100%	105%	97%	147%	99%	--[e]	79%	102%	107%	89%	104%	113%	11%	97%
Scheme 4, Replicate 2																	
Solution	0.03	4.09	0.013	1.44	0.020	0.050	2.66	0.023	--[e]	0.15	0.67	33.8	<0.20	<0.020	<0.20	6.80	0.089
Actual spike	0.935	23	0.063	25	1.82	1.72	4.61	5.01	--[e]	1.34	3.31	19.7	15.8	0.056	5.80	0.24	0.07
Theor. spike	1.25	25	0.050	25	1.66	1.66	3.33	5.00	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	75%	92%	126%	100%	109%	104%	138%	100%	--[e]	80%	99%	118%	95%	110%	116%	73%	30%
Scheme 4, Replicate 3																	
Solution	0.038	4.09	0.013	1.56	0.020	0.060	2.43	0.024	--[e]	0.13	0.59	33.8	<0.20	<0.020	<0.20	6.67	0.076
Actual spike	0.460	23	0.054	24	1.75	1.66	4.91	5.05	--[e]	1.35	3.31	20.8	15.5	0.056	5.55	0.27	0.18
Theor. spike	1.25	25	0.050	25	1.66	1.66	3.33	5.00	--[e]	1.67	3.33	16.6	16.6	0.050	5.00	0.33	0.25
% Recovery	37%	92%	106%	96%	105%	100%	147%	101%	--[e]	81%	99%	125%	93%	112%	110%	80%	72%

[a] All concentrations are in mg/L unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

[e] Insufficient sample to complete analysis; spike is being repeated.

APPENDIX C

RAW DATA FROM PHASE 1 ANALYSES OF FLY ASH

This appendix summarizes Phase 1 analyses performed on samples of fly ash. The Phase 1 procedures consisted of the following general analytical schemes:

Scheme 1 - EP test with leachate digestion

Scheme 2 - EP test without leachate digestion

Scheme 3 - digestion according to EPA (1979a) with sample stored at room temperature

Scheme 4 - digestion according to EPA (1979a) with sample preserved by addition of nitric acid (HNO_3) to pH <2

Results of determinations from Schemes 1 and 2 were used to evaluate the requirement to digest leachates resulting from the EP test. Results of determinations from Schemes 3 and 4 were used to evaluate sample storage requirements.

Raw data from the Phase 1 analyses of fly ash are presented in Table C-1. The mean and standard deviation for elemental determinations by ICP and AAS are summarized for each analytical scheme in Table C-2. Recoveries of metal spikes from sample digestates and leachates (after storage for two weeks) are presented in Table C-3.

TABLE C-1. RAW DATA FROM PHASE 1 ANALYSES OF FLY ASH

Element	Method	Concentration[a]											
		Scheme 1[b] (ERCO ID TM-81-1257)			Scheme 2[c] (ERCO ID TM-81-1258)			Scheme 3[d] (ERCO ID TM-81-1259)			Scheme 4[e]		
		Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3
Al	ICP	5.20	4.86	5.09	5.68	5.10	5.53	7,880	8,440	8,440	--	--	--
	AAS	<5.86	<5.22	<5.78	<5.56	<5.56	<5.56	8,250	8,590	8,320	--	--	--
Sb	ICP	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	2.0	2.0	1.7	--	--	--
	AAS	<0.080	<0.080	<0.080	<0.500	<0.500	<0.500	<10	<10	<10	--	--	--
As	ICP	<0.016	<0.016	<0.016	<0.080	<0.080	<0.080	43	40	44	--	--	--
	AAS	0.008	<0.008	<0.006	<0.010	<0.010	<0.010	32	31	38	--	--	--
Ba	ICP	0.020	0.007	0.006	0.138	0.167	0.189	129	135	133	--	--	--
	AAS	<0.040	<0.040	<0.040	<0.250	<0.250	<0.250	123	131	101	--	--	--
Be	ICP	0.007	0.006	0.006	0.006	0.006	0.007	2.3	2.5	2.5	--	--	--
	AAS	0.005	0.005	0.005	<0.025	<0.025	<0.025	2.6	2.0	1.9	--	--	--
Cd	ICP	0.006	0.006	0.006	0.007	0.007	0.008	<0.05	<0.05	<0.05	--	--	--
	AAS	0.006	0.006	0.006	<0.010	<0.010	<0.010	0.25	0.25	0.25	--	--	--
Cr	ICP	0.020	0.021	0.023	0.013	0.010	0.012	37	38	41	--	--	--
	AAS	<0.030	<0.030	<0.030	<0.180	<0.180	<0.180	34	39	45	--	--	--
Cu	ICP	0.458	0.476	0.475	0.461	0.450	0.450	28	25	26	--	--	--
	AAS	0.455	0.466	0.476	0.455	0.436	0.436	31	28	34	--	--	--
Fe	ICP	0.284	0.118	0.174	0.210	0.116	0.311	2,300	2,420	3,180	--	--	--
	AAS	0.302	0.136	0.188	0.222	0.168	0.376	2,900	2,900	3,700	--	--	--
Pb	ICP	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	13	14	15	--	--	--
	AAS	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	12	14	14	--	--	--
Mn	ICP	0.056	0.055	0.059	0.054	0.055	0.062	18	19	21	--	--	--
	AAS	0.053	0.055	0.058	0.055	0.055	0.068	17	18	20	--	--	--
Hg (ug/L)	ICP	<10	<10	<10	<10	<10	<10	<10	<10	<10	--	--	--
	AAS	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.014	0.014	0.013	--	--	--
Ni	ICP	0.046	0.045	0.045	0.045	0.038	038	10	11	12	--	--	--
	AAS	0.047	0.042	0.047	0.057	0.072	0.057	11	13	14	--	--	--
Se	ICP	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	<10[g]	<10[g]	<10[g]	--	--	--
	AAS	0.022	0.021	0.023	0.019	0.015	0.015	6.0	5.5	4.3	--	--	--
Ag	ICP	<0.001	<0.001	<0.001	<0.005	<0.005	<0.005	<0.500	<0.500	<0.500	--	--	--
	AAS	<0.005	<0.005	<0.005	<0.050	<0.050	<0.050	<0.500	<0.500	<0.500	--	--	--
Tl	ICP	<0.030	<0.030	<0.030	<0.150	<0.150	<0.150	<2.0	<2.0	<2.0	--	--	--
	AAS	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	<2.0	<2.0	<2.0	--	--	--
Zn	ICP	0.150	0.167	0.174	0.163	0.161	0.162	20	22	23	--	--	--
	AAS	0.138	0.148	0.164	0.143	0.143	0.143	24	25	24	--	--	--

[a] Unless otherwise noted, concentrations are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] No Scheme 4 analyses were performed on fly ash since preservation with HNO_3 is not applicable to this material.

[g] Severe interferences for this sample.

-- Not analyzed.

TABLE C-2. SUMMARY OF PHASE 1 ANALYSES OF FLY ASH

		Concentration[a]			
Element	Method	Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	5.05 ± 0.17	5.43 ± 0.30	8,253 ± 323	---
	AAS	5.62 ± 0.35	5.56 ± 0.00	8,387 ± 179	---
Sb	ICP	<0.010[f]	<0.050[f]	1.9 ± 0.12	---
	AAS	<0.080[f]	<0.080[f]	<10	---
As	ICP	<0.016[f]	<0.080[f]	42 ± 2.1	---
	AAS	0.007 ± 0.001	<0.010[f]	34 ± 3.8	---
Ba	ICP	0.011 ± 0.008	0.165 ± 0.026	132 ± 3.1	---
	AAS	<0.040[f]	<0.250[f]	118 ± 16	---
Be	ICP	0.006 ± 0.001	0.006 ± 0.001	2.46 ± 0.12	---
	AAS	0.005 ± 0.0	<0.025[f]	2.2 ± 0.38	---
Cd	ICP	0.006 ± 0.001	0.007 ± 0.001	<0.05	---
	AAS	0.006 ± 0.001	<0.010[f]	0.25 ± 0.00	---
Cr	ICP	0.021 ± 0.001	0.012 ± 0.001	39 ± 2.1	---
	AAS	<0.030[f]	<0.180[f]	39 ± 5.5	---
Cu	ICP	0.469 ± 0.010	0.454 ± 0.006	27 ± 1.5	---
	AAS	0.465 ± 0.010	0.442 ± 0.010	31 ± 3.0	---
Fe	ICP	0.192 ± 0.085	0.212 ± 0.097	2,630 ± 477	---
	AAS	0.209 ± 0.085	0.255 ± 0.108	3,166 ± 462	---
Pb	ICP	<0.010[f]	<0.010[f]	14 ± 1.0	---
	AAS	<0.040[f]	<0.200[f]	13 ± 1.2	---
Mn	ICP	0.057 ± 0.002	0.057 ± 0.005	19 ± 1.5	---
	AAS	0.055 ± 0.003	0.059 ± 0.007	18 ± 1.5	---
Hg	ICP	---	---	---	---
	AAS	<0.01 ug/l	<0.01 ug/l	0.014 ± 0.001	---
Ni	ICP	0.045 ± 0.001	0.041 ± 0.004	11 ± 1.0	---
	AAS	0.045 ± 0.003	0.062 ± 0.009	13 ± 1.5	---
Se	ICP	<0.010[f]	<0.050[f]	<10	---
	AAS	0.022 ± 0.001	0.016 ± 0.002	5.3 ± 0.87	---
Ag	ICP	<0.001[f]	<0.005[f]	<0.50 [f]	---
	AAS	<0.005[f]	<0.025[f]	<0.50 [f]	---
Ti	ICP	<0.030[f]	<0.150[f]	<2.0	---
	AAS	<0.040[f]	<0.200[f]	<2.0	---
Zn	ICP	0.164 ± 0.012	0.162 ± 0.001	22 ± 1.5	---
	AAS	0.150 ± 0.013	0.143 ± 0.001	24 ± 0.58	

---

[a] Mean and standard deviation; unless otherwise noted, units are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] No Scheme 4 analyses were performed on fly ash since preservation with HNO_3 is not applicable to this material.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

--- Not analyzed

TABLE C-3. SPIKE RECOVERIES FOR PHASE 1 ANALYSES OF FLY ASH[a]

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 1, Replicate 1																	
Solution[b]	<0.025	35	0.033	<0.25	0.027	0.038	<0.2	2.73	1.80	0.33	0.27	<0.25	<0.50	0.077	<0.20	0.83	0.077
Actual spike[c]	1.30	46	0.066	<0.25	2.57	2.48	5.68	4.95	5.60	2.69	5.00	--[e]	25	0.025	5.90	0.50	0.20
Theor. spike[d]	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	--[e]	25	0.050	5.00	0.50	0.25
% Recovery	10.4%	100%	131%	0%	103%	99%	113%	99%	112%	107%	100%	--[e]	100%	50%	118%	100%	80%
Scheme 1, Replicate 2																	
Solution	<0.025	31	0.024	<0.25	0.030	0.035	<0.2	2.80	0.82	0.35	0.25	<0.25	<0.50	0.071	<0.20	0.89	0.026
Actual spike	0.62	44	0.068	<0.25	2.55	2.46	5.52	4.94	4.88	2.68	5.06	18	26	0.018	5.67	0.41	0.23
Theor. spike	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	49%	96%	135%	0%	100%	98%	110%	99%	97%	107%	101%	72%	104%	35%	113%	82%	92%
Scheme 1, Replicate 3																	
Solution	<0.025	35	0.032	<0.25	0.030	0.034	<0.2	2.86	1.13	0.36	0.27	<0.25	<0.50	0.106	<0.20	0.99	0.045
Actual spike	1.31	43	0.056	<0.25	2.42	2.39	5.49	4.82	4.93	2.67	5.00	17	25	<0.002	5.80	0.39	0.22
Theor. spike	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	104%	93%	112%	0%	97%	96%	109%	96%	98%	107%	100%	68%	100%	<4%	116%	78%	88%

[a] All concentrations are in mg/L unless otherwise noted
[b] Solution concentration
[c] Measured spike concentration
[d] Theoretical spike concentration
[e] Insufficient sample to complete analysis
-- Not analyzed

Table C-3. Continued

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 2, Replicate 1																	
Solution[b]	<0.025	5.6	0.048	<0.5	<0.01	<0.01	<0.2	0.46	0.20	0.06	0.05	<0.25	<0.50	0.088	<0.20	0.78	0.02
Actual spike[c]	1.25	44	0.053	26	2.44	2.44	5.37	5.08	5.19	2.23	4.81	25	24	0.038	5.90	0.18	0.30
Theor. spike[d]	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	100%	96%	106%	104%	98%	98%	107%	102%	104%	89%	96%	100%	96%	75%	118%	35%	122%
Scheme 2, Replicate 2																	
Solution	<0.025	5.6	0.028	<0.5	<0.01	<0.01	<0.2	0.44	0.12	0.07	0.05	<0.25	<0.50	0.075	<0.20	0.89	0.04
Actual spike	1.33	44	0.055	33	2.44	2.46	5.49	5.08	5.44	2.25	4.81	25	24	0.037	5.50	0.11	0.27
Theor. spike	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	106%	96%	110%	132%	98%	98%	109%	102%	108%	90%	96%	100%	96%	73%	110%	23%	107%
Scheme 2, Replicate 3																	
Solution	<0.025	5.6	0.022	<0.5	<0.01	<0.01	<0.2	0.44	0.37	0.07	0.05	<0.25	<0.50	0.084	<0.20	0.83	0.02
Actual spike	1.33	45	0.066	22	2.44	2.41	5.41	4.98	5.51	2.27	4.81	26	24	0.027	5.40	0.15	0.28
Theor. spike	1.25	46	0.050	25	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	106%	98%	131%	88%	98%	96%	108%	99%	110%	91%	96%	104%	96%	54%	108%	30%	112%

[a] All concentrations are in mg/L unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

Table C-3. Continued

Sample Identification	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (ug)
Scheme 3, Replicate 1																	
Solution[b]	<0.025	10	0.039	0.15	0.16	0.031	4.24	3.87	3.62	2.16	1.41	1.5	<0.50	0.006	<0.20	2.94	0.01
Actual spike[c]	1.25	42	0.063	47	2.44	2.36	6.29	4.[illegible]	4.54	2.97	5.10	25	25	0.048	5.80	0.29	0.27
Theor. spike[d]	1.25	50	0.050	50	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	100%	84%	125%	94%	98%	94%	126%	98%	91%	119%	102%	100%	100%	96%	116%	58%	108%
Scheme 3, Replicate 2																	
Solution	<0.025	9.3	0.033	0.14	0.18	0.026	4.24	3.05	3.18	1.96	1.37	1.5	<0.50	0.006	<0.20	2.64	0.01
Actual spike	1.25	41	0.061	47	2.40	2.39	7.21	4.82	4.80	2.90	5.36	25	25	0.046	5.80	0.38	0.26
Theor. spike	1.25	50	0.050	50	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	100%	82%	123%	94%	96%	96%	144%	96%	96%	116%	107%	100%	100%	92%	116%	76%	104%
Scheme 3, Replicate 3																	
Solution	<0.025	8.3	0.038	0.11	0.18	0.034	4.51	3.36	3.75	1.99	1.33	1.4	<0.50	0.005	<0.20	2.40	0.01
Actual spike	1.36	41	0.091	48	2.40	2.42	5.77	4.66	4.71	2.94	5.14	25	24	0.049	6.10	0.38	0.26
Theor. spike	1.25	50	0.050	50	2.50	2.50	5.00	5.00	5.00	2.50	5.00	25	25	0.050	5.00	0.50	0.25
% Recovery	108%	82%	182%	96%	96%	97%	115%	93%	94%	117%	103%	100%	96%	97%	122%	76%	105%

[a] All concentrations are in mg/L unless otherwise noted.

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

APPENDIX D

RAW DATA FROM PHASE 1 ANALYSES OF LOW-pH SLUDGE

This appendix summarizes Phase 1 analyses performed on samples of low-pH sludge. The Phase 1 procedures consisted of the following general analytical schemes:

Scheme 1 - EP test with leachate digestion

Scheme 2 - EP test without leachate digestion

Scheme 3 - digestion according to EPA (1979a) with sample stored at room temperature

Scheme 4 - digestion according to EPA (1979a) with sample preserved by addition of nitric acid (HNO_3) to pH <2

Results of determinations from Schemes 1 and 2 were used to evaluate the requirement to digest leachates resulting from the EP test. Results of determinations from Schemes 3 and 4 were used to evaluate sample storage requirements.

Raw data from the Phase 1 analyses of low-pH sludge are presented in Table D-1. The mean and standard deviation for elemental determinations by ICP and AAS are summarized for each analytical scheme in Table D-2. Spike recoveries for each sample digestate and leachate (after storage within two weeks) are summarized in Table D-3.

TABLE D-1. RAW DATA FROM PHASE I ANALYSES OF LOW-pH SLUDGE

		Concentration[a]											
		Scheme 1[b]			Scheme 2[c]			Scheme 3[d]			Scheme 4[e]		
Element	Method	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3
Al	ICP	14	15	16	15	15	15	1780	1950	1800	2000	1870	1780
	AAS	16	16	15	14	15	15	1680	2040	1880	2100	1950	1840
Sb	ICP	<0.053	<0.053	<0.053	<0.200	<0.200	<0.200	<56	<53	<49	<54	<57	<54
	AAS	<0.053	<0.053	<0.053	<0.200	<0.200	<0.200	<2.2	<2.1	<1.9	<2.2	<2.3	<2.1
As	ICP	<2.0[f]	<2.0[f]	<2.0[f]	0.520	0.468	0.422	<23	<18	<20	<21	<23	<21
	AAS	0.065	0.048	0.056	0.098	0.089	0.089	1.8	2.0	1.8	2.1	2.1	1.9
Ba	ICP	0.168	0.133	0.151	0.145	0.109	0.123	21	22	23	21	29	26
	AAS	<0.500	<0.500	<0.500	<0.500	0.510	<0.500	25	26	28	20	26	26
Be	ICP	<0.003	<0.003	<0.003	<0.010	<0.010	<0.010	<2.8	<2.7	<2.4	<2.7	<2.8	<2.7
	AAS	<0.013	<0.013	<0.013	<0.010	<0.010	<0.010	<0.110	<0.110	<0.100	<0.110	<0.110	<0.110
Cd	ICP	0.34	0.33	0.30	0.28	0.33	0.23	2.6	2.7	2.7	2.5	2.3	2.7
	AAS	0.24	0.25	0.24	0.23	0.23	0.22	2.7	2.7	2.7	2.5	2.6	2.5
Cr	ICP	15	14	15	15	15	15	1380	1380	1380	1330	1340	1340
	AAS	16	17	15	17	17	16	1370	1380	1330	1380	1320	1380
Cu	ICP	2.3	2.5	2.3	2.7	2.8	2.4	37	37	36	35	34	34
	AAS	2.3	2.5	2.2	2.1	2.3	2.0	32	32	31	32	32	32
Fe	ICP	1610	1590	1500	1610	1610	1550	29,200	29,300	29,100	27,700	27,800	27,600
	AAS	1580	1630	1510	1590	1610	1530	28,900	29,100	28,800	28,400	28,400	28,600
Pb	ICP	5.1	5.5	4.7	4.3	4.9	4.6	138	139	134	120	127	132
	AAS	4.2	4.3	4.1	4.1	4.1	4.0	117	116	118	115	117	117
Mn	ICP	30	29	31	30	30	29	381	381	381	367	368	369
	AAS	30	33	31	30	31	30	382	380	379	372	375	369
Hg	ICP	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
	AAS	<0.0001	0.0002	<0.0001	<0.0001	<0.0001	<0.0001	0.13	0.090	0.084	0.097	0.10	0.10
Ni	ICP	76	75	79	75	75	72	954	967	960	913	923	935
	AAS	82	83	79	75	75	74	940	942	940	928	936	928
Se	ICP	<50[f]	<50[f]	<50[f]	<11[f]	<11[f]	<11[f]	<343[f]	<275[f]	<307[f]	<318[f]	<347[f]	<312[f]
	AAS	<0.050[f]	<0.050[f]	<0.050[f]	<0.050[f]	<0.050[f]	<0.050[f]	<0.57[f]	<0.46[f]	<0.51[f]	<0.53[f]	<0.58[f]	<0.52[f]
Ag	ICP	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.92	<0.96	<1.05	<0.98	<1.16	<1.04
	AAS	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.23	<0.24	<0.24	<0.24	<0.29	<0.26
Tl	ICP	<5.0	<5.0	<5.0	<1.0	<1.0	<1.0	<46	<48	<52	<49	<58	<52
	AAS	<0.080	<0.080	<0.080	<0.200	<0.200	<0.200	<1.8	<1.9	<1.9	<2.0	<2.3	<2.1
Zn	ICP	3750	3790	3480	3530	3540	3310	41,300	40,500	40,100	40,200	39,800	39,500
	AAS	3930	4100	3810	3180	3200	3030	40,800	39,600	39,400	39,100	38,800	39,500

[a] Mean and standard deviation; unless otherwise noted, units are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

-- Not analyzed.

TABLE D-2. SUMMARY OF PHASE 1 ANALYSES OF LOW-pH SLUDGE

Element	Method	Concentration[a]			
		Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	15±1	15±0	1840±90	1880±110
	AAS	16±1	15±1	1870±180	1960±130
Sb	ICP	<0.053[f]	<0.200[f]	<49[f]	<54[f]
	AAS	<0.053[f]	<0.200[f]	<1.9[f]	<2.1[f]
As	ICP	<2.0[f]	0.470±0.049	<18[f]	<21[f]
	AAS	0.056±0.009	0.092±0.005	1.9±0.1	2.0±0.1
Ba	ICP	0.151±0.018	0.126±0.018	22±1	25±4
	AAS	<0.500[f]	<0.500[f]	26±2	24±3
Be	ICP	<0.003[f]	<0.010[f]	<2.4[f]	<2.7[f]
	AAS	<0.013[f]	<0.010[f]	<0.100[f]	<0.110[f]
Cd	ICP	0.32±0.02	0.28±0.05	2.7±0.1	2.5±0.2
	AAS	0.24±0.01	0.23±0.01	2.7±0	2.5±0.1
Cr	ICP	15±1	15±0	1380±0	1340±10
	AAS	16±1	17±1	1360±30	1360±40
Cu	ICP	2.4±0.1	2.6±0.2	37±1	34±1
	AAS	2.3±0.2	2.1±0.2	32±1	32±0
Fe	ICP	1570±60	1590±30	29,200±100	27,700±100
	AAS	1570±60	1580±40	28,900±200	28,500±100
Pb	ICP	5.1±0.4	4.6±0.3	137±3	126±6
	AAS	4.2±0.1	4.1±0.1	117±1	116±1
Mn	ICP	30±1	30±1	381±0	368±1
	AAS	31±2	30±1	380±2	372±3
Hg	ICP	--	--	--	--
	AAS	<0.0001[f]	<0.0001[f]	0.10±0.03	0.10±0
Ni	ICP	77±2	74±2	960±7	924±11
	AAS	81±2	75±1	941±1	931±5
Se	ICP	<50[f]	<11[f]	<275[f]	<312[f]
	AAS	<0.050[f]	<0.050[f]	<0.46[f]	<0.52[f]
Ag	ICP	<0.100[f]	<0.010[f]	<0.92[f]	<0.98[f]
	AAS	<0.010[f]	<0.010[f]	<0.23[f]	<0.24[f]
Tl	ICP	<5.0[f]	<1.0[f]	<46[f]	<49[f]
	AAS	<0.080[f]	<0.200[f]	<1.8[f]	<2.0[f]
Zn	ICP	3670±170	3460±130	40,600±600	39,800±400
	AAS	3950±150	3140±90	39,900±800	39,100±400

[a] Mean and standard deviation; unless otherwise noted, units are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion.

[c] EP toxicity test without leachate digestion.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] Digestion according to EPA[2]. Sample preserved with HNO_3 to pH <2.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

-- Not analyzed.

TABLE D-3. SPIKE RECOVERIES FOR PHASE I ANALYSES OF LOW-pH SLUDGE[a]

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg/g)
Scheme 1, Replicate 1																	
Solution[b]	<0.010	1.6	0.032	0.25	<0.003	0.18	12.1	1.72	1175	22.6	61	3.1	<0.20	<0.125	<0.200	2940	<0.01
Actual spike[c]	0.126	18.7	0.165	<1.00	0.82	1.01	22.3	4.71	1039	51.8	87	2.55	9.7	0.174	5.11	988	0.24
Theor. spike[d]	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery	25	94	83	0	82	101	111	94	104	104	87	51	97	87	102	99	96
Scheme 1, Replicate 2																	
Solution	<0.010	12.2	0.024	0.28	<0.003	0.19	12.7	1.90	1220	24.4	62	3.2	<0.20	<0.125	<0.200	3080	<0.01
Actual spike	0.147	18.6	0.169	<1.00	0.78	0.94	21.2	4.49	902	46.9	81	1.75	9.5	0.188	5.22	653	0.24
Theor. spike	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery	29	93	85	0	78	94	106	90	90	94	81	35	95	94	104	65	96
Scheme 1, Replicate 3																	
Solution	<0.010	11.4	0.028	0.28	<0.003	0.18	11.3	1.64	1130	23.4	59	3.1	<0.20	<0.125	<0.200	2850	<0.01
Actual spike	0.062	19.4	0.165	<1.00	0.85	1.00	23.1	4.83	1053	49.1	87	1.80	9.9	0.188	5.00	937	0.24
Theor. spike	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery	12	97	83	0	85	100	115	97	105	98	87	36	99	94	100	94	96

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[a] All concentrations are in mg/L in solution except mercury which is in units of µg/g of sludge.

[b] Solution concentration.

[c] Measured spike concentration.

[d] Theoretical spike concentration.

Table D-3 (continued)

Sample Identification	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg/g)
Scheme 2, Replicate 1																	
Solution[b]	<0.010	8.0	0.094	<0.500	<0.013	0.13	9.7	1.21	917	17.6	44	2.34	<0.200	<0.050	<0.200	1840	<0.01
Actual spike[c]	0.126	18.8	0.106	<1.0	0.90	1.00	21.3	4.88	942	49.5	90	4.67	9.9	0.155	5.17	1254	0.24
Theor. spike[d]	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery[e]	25	94	53	0	90	100	107	98	94	99	90	93	99	78	103	125	96
Scheme 2, Replicate 2																	
Solution	<0.010	8.5	0.085	<0.500	<0.013	0.13	10.0	1.33	929	17.9	44	2.38	<0.200	<0.050	<0.200	1850	<0.01
Actual spike	0.112	19.3	0.115	<1.0	0.89	0.99	21.2	4.84	904	49.8	91	4.49	9.8	0.155	5.11	1266	0.24
Theor. spike	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery	22	97	58	0	89	99	106	97	90	100	91	90	98	78	102	127	96
Scheme 2, Peplicate 3																	
Solution	<0.010	8.4	0.085	<0.500	<0.013	0.13	9.2	1.16	885	17.1	42	2.29	<0.200	<0.050	<0.200	1750	<0.01
Actual spike	0.126	18.6	0.115	<1.0	0.90	0.97	21.1	4.82	922	49.7	92	4.16	9.7	0.155	5.28	1322	0.24
Theor. spike	0.500	20.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	50.0	100	5.00	10.0	0.200	5.00	1000	0.25
% Recovery	25	93	58	0	90	97	106	96	92	99	92	83	97	78	106	132	96

[a] All concentrations are in mg/L in solution except mercury which is in units of ug/g of sludge.

[b] Solution concentration.

[c] Measured spike concentration.

[d] Theoretical spike concentration.

Table D-3 (continued)

Sample Identification	Element Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg/g)
Scheme 3, Replicate 1																	
Solution[b]	<0.025	30.0	0.012	0.45	<0.010	0.048	24.4	0.57	517	6.8	16.8	2.08	<0.200	<0.050	<0.200	729	0.10
Actual spike[c]	0.034	53.6	0.186	<1.0	0.92	0.98	22.3	4.91	952	10.3	18.3	2.64	9.5	0.195	5.28	980	0.057
Theor. spike[d]	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.063
% Recovery	7	107	93	0	92	98	111	98	95	103	92	53	95	98	106	98	90
Scheme 3, Replicate 2																	
Solution	<0.025	38.2	0.043	0.49	<0.010	0.050	25.8	0.60	545	7.1	17.7	2.18	<0.200	<0.050	<0.200	742	0.10
Actual spike	0.034	49.2	0.179	<1.0	0.93	0.98	22.7	4.94	917	10.1	17.4	2.63	9.6	0.195	5.22	998	0.029
Theor. spike	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.49
% Recovery	7	98	90	0	93	98	113	99	94	101	87	53	96	98	104	100	59
Scheme 3, Replicate 3																	
Solution	<0.025	38.8	0.036	0.58	<0.010	0.055	27.4	0.65	593	7.8	19.4	2.44	<0.200	<0.050	<0.200	811	0.10
Actual spike	0.041	42.9	0.186	<1.0	0.93	0.98	22.9	4.92	954	9.9	17.1	2.51	9.7	0.195	5.17	989	0.036
Theor. spike	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.037
% Recovery	8	86	93	0	93	98	114	98	95	99	86	50	97	98	103	99	103

[a] All concentrations are in mg/L in solution except mercury which is in units of µg/g of sludge.
[b] Solution concentration.
[c] Measured spike concentration.
[d] Theoretical spike concentration.

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Table D-3 (continued)

Sample Identification	Element Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (μg/g)
Scheme 4, Replicate 1																	
Solution[b]	<0.025	38.8	0.019	0.37	<0.010	0.046	25.4	0.59	525	6.9	17.1	2.12	<0.200	<0.050	<0.200	722	0.10
Actual spike[c]	0.055	44.8	0.176	<1.0	0.94	0.98	22.2	4.91	944	10.0	17.7	2.46	9.7	0.181	5.06	967	0.063
Theor. spike[d]	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.076
% Recovery	11	90	88	0	94	98	111	98	94	100	89	49	97	91	101	97	83
Scheme 4, Replicate 2																	
Solution	<0.025	34.2	0.036	0.46	<0.010	0.045	21.2	0.57	500	6.6	16.4	2.06	<0.200	<0.050	<0.200	682	0.10
Actual spike	0.041	47.5	0.179	<1.0	0.90	1.00	24.8	4.89	941	10.0	18.0	2.56	9.8	0.181	5.06	987	0.094
Theor. spike	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.069
% Recovery	8	95	90	0	90	100	124	98	94	100	90	51	98	91	101	99	136
Scheme 4, Replicate 3																	
Solution	<0.025	34.2	0.036	0.49	<0.010	0.047	25.8	0.59	514	6.9	17.3	2.18	<0.200	<0.050	<0.200	735	0.10
Actual spike	0.041	41.6	0.175	<1.0	0.89	0.98	21.3	4.87	961	10.1	17.1	3.10	9.8	0.202	5.06	974	0.050
Theor. spike	0.500	50.0	0.200	10.0	1.00	1.00	20.0	5.00	1000	10.0	20.0	5.00	10.0	0.200	5.00	1000	0.061
% Recovery	8	87	88	0	89	98	107	97	96	101	86	62	98	101	101	97	82

[a] All concentrations are in mg/L in solution except mercury which is in units of μg/g of sludge.

[b] Solution concentration.

[c] Measured spike concentration.

[d] Theoretical spike concentration.

APPENDIX E

RAW DATA FROM PHASE 1 ANALYSES OF OIL/WATER WASTE

This appendix summarizes Phase 1 analyses performed on the aqueous and solid phases of the oil/water waste. The Phase 1 procedures consisted of the following general analytical schemes:

Scheme 1 - EP test with leachate digestion

Scheme 2 - EP test without leachate digestion

Scheme 3 - digestion according to EPA (1979a) with sample stored at room temperature

Scheme 4 - digestion according to EPA (1979a) with sample preserved by addition of nitric acid (HNO_3) to pH <2

Results of determinations from Schemes 1 and 2 were used to evaluate the requirement to digest leachates resulting from the EP test. Results of determinations from Schemes 3 and 4 were used to evaluate sample storage requirements.

In addition, the oil phase was extracted and analyzed separately in Schemes 1 and 2 to enable estimation of the metal partitioning between organic and inorganic phases.

Raw data from the Phase 1 analyses of the aqueous and solid phases of the oil/water waste are presented in Table E-1. The mean and standard deviation for elemental determinations by ICP and AAS are summarized for each analytical scheme in Table E-2. Spike recoveries for each aqueous sample digestate and leachate (after storage for 2 weeks) are summarized in Table E-3.

TABLE E-1. RAW DATA FROM PHASE 1 ANALYSES OF OIL/WATER WASTE

Element	Method	Concentration[a]											
		Scheme 1[b]			Scheme 2[c]			Scheme 3[d]			Scheme 4[e] -		
		Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3
Al	ICP	0.111	0.211	0.112	0.094	<0.050	0.164	8100	7210	8630	9000	7710	6740
	AAS	0.098	0.225	0.153	<0.500	<0.500	<0.500	7860	6820	8230	8850	7580	6860
Sb	ICP	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	<20	<16	<20	<18	<17	<19
	AAS	<0.100	<0.100	<0.100	<0.500	<0.500	<0.500	<50[f]	<41[f]	<49[f]	<45[f]	<44[f]	<47[f]
As	ICP	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	<19[f]	<19[f]	<18[f]	<17[f]	<17[f]	<17[f]
	AAS	0.003	0.003	0.004	<0.005	<0.005	<0.005	6.4	7.8	6.2	5.4	7.5	8.7
Ba	ICP	0.017	0.022	0.013	0.039	<0.010	0.017	84	88	89	105	82	73
	AAS	<0.100	<0.100	<0.100	<0.500	<0.500	<0.500	72	74	87	90	66	59
Be	ICP	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	<1.00	<0.82	<0.98	<0.90	<0.87	<0.94
	AAS	<0.001	<0.001	<0.001	<0.005	<0.005	<0.005	0.36	0.32	0.39	0.39	0.38	0.38
Cd	ICP	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	<1.00	<0.82	<0.88	<0.90	<0.87	<0.94
	AAS	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	0.76	0.71	0.76	0.99	1.1	0.77
Cr	ICP	<0.005	<0.005	<0.005	<0.025	<0.025	<0.025	25	20	26	24	22	23
	AAS	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	29	23	31	30	27	27
Cu	ICP	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	24	42	22	24	36	27
	AAS	<0.004	<0.004	<0.004	<0.020	<0.020	<0.020	16	25	20	20	30	21
Fe	ICP	0.396	0.475	0.478	1.01	0.36	0.99	14,300	12,000	14,800	15,100	14,800	13,300
	AAS	0.402	0.485	0.486	1.00	0.40	0.70	14,800	12,500	15,300	15,500	15,500	13,500
Pb	ICP	<0.040	<0.040	<0.040	<0.200	<0.200	<0.200	85	105	127	122	81	112
	AAS	<0.020	<0.020	<0.020	<0.100	<0.100	<0.100	79	91	115	116	66	104
Mn	ICP	0.079	0.088	0.074	0.122	0.053	0.077	108	78	84	85	82	73
	AAS	0.085	0.091	0.079	0.137	0.054	0.060	110	76	85	86	80	75
Hg	ICP	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
	AAS	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.014	0.006	0.011	0.010	0.008	0.010
Ni	ICP	0.017	0.012	0.012	<0.050	<0.050	<0.050	12	12	12	12	11	9
	AAS	<0.010	<0.010	<0.010	<0.050	<0.050	<0.050	11	11	11	12	10	10
Se	ICP	<0.020	<0.020	<0.020	<0.100	<0.100	<0.100	<190[f]	<190[f]	<180[f]	<170[f]	<170[f]	<170[f]
	AAS	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.94	<0.95	<0.90	<0.84	<0.87	<0.87
Ag	ICP	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	<0.085	<0.098	<0.090	<0.099	<0.086	<0.094
	AAS	<0.002	<0.002	<0.002	<0.010	<0.010	<0.010	<0.085	<0.098	<0.090	<0.099	<0.086	<0.094
Tl	ICP	<0.100	<0.100	<0.100	<0.500	<0.500	<0.500	<43[f]	<49[f]	<45[f]	<50[f]	<43[f]	<47[f]
	AAS	<0.020	<0.020	<0.020	<0.100	<0.100	<0.100	<0.85	<0.98	<0.90	<0.99	<0.86	<0.94
Zn	ICP	0.088	0.100	0.073	0.111	0.031	0.045	94	110	110	111	99	116
	AAS	0.083	0.093	0.071	0.111	0.025	0.025	89	100	100	100	95	110

[a] Unless otherwise noted, concentrations are mg/L for Schemes 1 and 2; ug/g for Schemes 3 and 4.

[b] EP toxicity test with leachate digestion; aqueous phase only.

[c] EP toxicity test without leachate digestion; aqueous phase only.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] Digestion according to EPa[2]. Sample preserved with HNO3 to pH <2.

[f] Severe interferences for this sample.

-- Not analyzed.

TABLE E-2. SUMMARY OF PHASE 1 ANALYSES OF OIL/WATER WASTE

		Concentration[a]			
Element	Method	Scheme 1[b]	Scheme 2[c]	Scheme 3[d]	Scheme 4[e]
Al	ICP	0.145 ± 0.057	0.103 ± 0.058	7980 ± 720	7820 ± 1100
	AAS	0.159 ± 0.064	<0.500[f]	7640 ± 730	7760 ± 1000
Sb	ICP	<0.040[f]	<0.200[f]	<16[f]	<17[f]
	AAS	<0.100[f]	<0.500[f]	<41[f]	<44[f]
As	ICP	<0.040[f]	<0.200[f]	<18[f]	<17[f]
	AAS	0.003 ± 0.001	<0.005[f]	6.8 ± 0.9	7.2 ± 1.7
Ba	ICP	0.017 ± 0.005	0.022 ± 0.015	87 ± 3	87 ± 17
	AAS	<0.100[f]	<0.500[f]	78 ± 8	72 ± 16
Be	ICP	<0.002[f]	<0.010[f]	<0.82[f]	<0.87[f]
	AAS	<0.001[f]	<0.005[f]	0.36 ± 0.04	0.38 ± 0.01
Cd	ICP	<0.002[f]	<0.010[f]	<0.82[f]	<0.87[f]
	AAS	<0.002[f]	<0.010[f]	0.74 ± 0.03	0.95 ± 0.17
Cr	ICP	<0.005[f]	<0.025[f]	24 ± 3	23 ± 1
	AAS	<0.010[f]	<0.050[f]	28 ± 4	28 ± 2
Cu	ICP	<0.010	<0.050	29 ± 11	29 ± 6
	AAS	<0.004[f]	<0.020[f]	20 ± 5	24 ± 6
Fe	ICP	0.450 ± 0.047	0.787 ± 0.370	13,700 ± 1490	14,400 ± 960
	AAS	0.458 ± 0.048	0.700 ± 0.300	14,200 ± 1490	14,830 ± 1150
Pb	ICP	<0.040[f]	<0.200[f]	106 ± 21	105 ± 21
	AAS	<0.020[f]	<0.100[f]	95 ± 18	95 ± 26
Mn	ICP	0.080 ± 0.007	0.084 ± 0.035	90 ± 16	80 ± 6
	AAS	0.085 ± 0.006	0.084 ± 0.046	90 ± 18	80 ± 6
Hg	ICP	<10	<10	<10	<10
	AAS	<0.001[f]	<0.001[f]	0.010 ± 0.004	0.009 ± 0.001
Ni	ICP	0.014 ± 0.003	<0.050[f]	12 ± 0	11 ± 2
	AAS	<0.010[f]	<0.050[f]	11 ± 0	11 ± 1
Se	ICP	<0.020	<0.100	<180[f]	<170[f]
	AAS	<0.005[f]	<0.005[f]	<0.090[f]	<0.084[f]
Ag	ICP	<0.002[f]	<0.010[f]	<0.085[f]	<0.086[f]
	AAS	<0.002[f]	<0.010[f]	<0.085[f]	<0.086[f]
Tl	ICP	<0.100[f]	<0.500[f]	<43[f]	<43[f]
	AAS	<0.020[f]	<0.100[f]	<0.85[f]	<0.86[f]
Zn	ICP	0.087 ± 0.014	0.062 ± 0.043	105 ± 9	109 ± 9
	AAS	0.082 ± 0.011	0.043 ± 0.048	96 ± 6	102 ± 8

[a] Mean and standard deviation; unless otherwise noted, units are mg/L for Schemes 1 and 2; ug/g for Scheme 3.

[b] EP toxicity test with leachate digestion; aqueous phase only.

[c] EP toxicity test without leachate digestion; aqueous phase only.

[d] Digestion according to EPA[2]. Sample stored at room temperature.

[e] Digestion according to EPA_2. Sample preserved with HNO_3 to pH <2.

[f] Mean and standard deviation were not calculated since one or more analyses were below the limit of detection.

-- Not analyzed.

TABLE E-3. SPIKE RECOVERIES FOR PHASE 1 ANALYSES OF OIL/WATER WASTE[a]

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg)
Scheme 1, Replicate 1																	
Solution[b]	<0.010	0.294	0.007	<0.100	<0.005	<0.005	<0.050	<0.020	1.20	0.253	<0.050	<0.100	<0.200	<0.010	<0.100	0.246	<0.01
Actual spike[c]	0.511	4.93	0.115	5.25	5.02	5.65	5.58	5.05	5.23	5.18	5.21	5.27	5.72	0.124	5.17	5.16	0.26
Theor. spike[d]	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	102	99	115	105	100	103	112	101	105	104	104	101	104	124	103	103	104
Scheme 1, Replicate 2																	
Solution	<0.010	0.673	0.009	<0.100	<0.005	<0.005	<0.050	<0.020	1.45	0.274	<0.050	<0.100	<0.200	<0.010	<0.100	0.280	<0.01
Actual spike	0.521	4.82	0.120	5.04	5.02	5.58	5.37	5.05	5.66	5.16	5.27	5.27	5.77	0.118	5.14	5.08	0.26
Theor. spike	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	104	96	120	101	100	101	107	101	113	103	105	101	104	118	103	102	104
Scheme 1, Replicate 3																	
Solution	<0.010	0.456	0.009	<0.100	<0.005	<0.005	<0.050	<0.020	1.45	0.235	<0.050	<0.100	<0.200	<0.010	<0.100	0.212	<0.01
Actual spike	0.511	4.63	0.098	4.97	4.99	5.65	4.97	5.05	5.25	5.07	5.21	5.15	5.62	0.115	5.14	5.11	0.25
Theor. spike	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	102	93	98	99	100	103	99	101	105	101	104	99	102	115	103	102	100

[a] All concentrations are in mg/L unless otherwise noted
[b] Solution concentration
[c] Measured spike concentration
[d] Theoretical spike concentration

Table E-3. Continued.

Sample Identification	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg)
Scheme 2, Replicate 1																	
Solution[b]	<0.010	<0.500	<0.005	<0.500	<0.005	<0.010	<0.050	<0.020	0.500	0.069	<0.050	<0.100	<0.500	<0.005	<0.100	0.056	<0.01
Actual spike[c]	0.502	4.96	0.104	5.25	5.02	5.71	5.17	5.05	5.13	5.23	5.14	5.21	5.62	0.109	5.04	5.07	0.26
Theor. spike[d]	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	100	99	104	105	100	104	103	101	103	105	103	100	102	109	101	101	104
Scheme 2, Replicate 2																	
Solution	<0.010	<0.500	<0.005	<0.500	<0.005	<0.010	<0.050	<0.020	0.200	0.027	<0.050	<0.100	<0.500	<0.005	<0.100	0.013	<0.01
Actual spike	0.507	4.96	0.101	4.97	4.93	5.52	5.17	5.05	5.16	5.15	5.17	5.15	5.57	0.109	5.10	5.04	0.26
Theor. spike	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	101	99	101	99	99	100	103	101	103	103	103	99	101	109	102	101	104
Scheme 2, Replicate 3																	
Solution	<0.010	<0.500	<0.005	<0.500	<0.005	<0.010	<0.050	<0.020	0.350	0.030	<0.050	<0.100	<0.500	<0.005	<0.100	0.013	<0.01
Actual spike	0.497	4.96	0.098	5.04	4.96	5.45	5.17	5.05	5.15	5.15	5.17	5.15	5.67	0.111	5.01	5.12	0.26
Theor. spike	0.500	5.00	0.100	5.00	5.00	5.50	5.00	5.00	5.00	5.00	5.00	5.20	5.50	0.100	5.00	5.00	0.25
% Recovery	99	99	98	101	99	99	103	101	103	103	103	99	103	111	100	102	104

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[a] All concentrations are in mg/L unless otherwise noted

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

Table B-3. Continued.

Sample Identification	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (μg)
	Element																
Scheme 3, Replicate 1																	
Solution[b]	<0.010	392	0.343	3.59	0.018	0.038	1.45	0.81	740	5.42	0.53	3.95	<5.0	<0.100	<0.100	4.43	0.014
Actual spike[c]	0.549	645	4.85	6.23	4.91	5.61	5.14	5.15	505	5.30	5.18	5.49	5.57	1.35	5.48	5.12	0.060
Theor. spike[d]	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	110	129	97	125	98	102	103	103	101	106	104	106	101	96	110	102	25
Scheme 3, Replicate 2																	
Solution	<0.010	414	0.410	4.48	0.020	0.043	1.42	1.51	760	4.61	0.67	5.51	<5.0	<0.100	<0.100	6.35	0.006
Actual spike	0.542	610	5.25	5.91	4.94	5.61	5.17	5.18	485	5.37	5.17	5.52	5.62	1.45	5.42	5.01	0.031
Theor. spike	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	108	122	105	118	99	102	103	104	97	107	103	106	102	104	108	100	13
Scheme 3, Replicate 3																	
Solution	<0.010	419	0.343	4.43	0.020	0.039	1.58	1.02	780	4.31	0.56	5.86	<5.0	<0.100	<0.100	5.28	0.011
Actual spike	0.547	605	5.39	5.74	4.82	5.61	4.81	5.12	631	5.05	5.15	5.41	5.62	1.45	5.48	4.73	0.016
Theor. spike	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	109	121	108	115	96	102	96	102	126	101	103	104	102	104	110	95	7

[a] All concentrations are in mg/L unless otherwise noted

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

Table E-3. Continued.

Sample Identification	Element																
	Ag	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Sb	Se	Tl	Zn	Hg (µg)
Scheme 4, Replicate 1																	
Solution[b]	<0.010	490	0.320	4.97	0.022	0.055	1.64	1.12	860	4.76	0.66	6.40	<5.0	<0.100	<0.100	5.77	0.010
Actual spike[c]	0.549	547	5.26	5.42	4.69	5.47	5.16	5.20	556	5.10	5.12	5.55	5.91	1.45	5.58	5.09	0.041
Theor. spike[d]	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	110	109	105	108	94	99	103	104	111	102	102	107	107	104	112	102	17
Scheme 4, Replicate 2																	
Solution	<0.010	436	0.432	3.82	0.022	0.061	1.54	1.73	890	4.61	0.56	3.78	<5.0	<0.100	<0.100	5.44	0.008
Actual spike	0.554	641	5.23	5.58	4.85	5.72	5.26	5.32	505	5.49	5.22	5.66	5.77	1.45	5.48	4.91	0.068
Theor. spike	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	111	128	105	112	97	104	105	106	101	110	104	109	105	104	110	98	28
Scheme 4, Replicate 3																	
Solution	<0.010	365	0.499	3.17	0.020	0.041	1.45	1.12	720	4.01	0.54	5.54	<5.0	<0.100	<0.100	5.98	0.010
Actual spike	0.544	606	5.16	5.10	4.88	5.61	5.14	5.39	541	5.11	5.20	5.61	5.82	1.65	5.42	4.88	0.030
Theor. spike	0.500	500	5.00	5.00	5.00	5.50	5.00	5.00	500	5.00	5.00	5.20	5.50	1.40	5.00	5.00	0.24
% Recovery	109	121	103	102	98	102	103	108	108	102	104	108	106	118	108	98	13

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[a] All concentrations are in mg/L unless otherwise noted

[b] Solution concentration

[c] Measured spike concentration

[d] Theoretical spike concentration

TABLE E-4. RAW DATA FROM PHASE 1 ANALYSES OF OIL PHASE OF OIL/WATER WASTE

		Concentration[a]					
		Unfiltered Scheme 1[b]			Filtered Scheme 2[c]		
Element	Method	Replicate 1	Replicate 2	Replicate 3	Replicate 1	Replicate 2	Replicate 3
Al	ICP	0.077	0.069	0.077	0.015	0.012	0.012
	AAS	--	--	--	--	--	--
Sb	ICP	<0.015	<0.015	<0.077	<0.015	<0.015	<0.016
	AAS	--	--	--	--	--	--
As	ICP	<0.021	<0.019	<0.021	<0.020	<0.020	<0.021
	AAS	<0.0051	<0.0049	<0.0051	<0.0050	<0.0051	<0.0053
Ba	ICP	0.0018	0.0019	0.0018	0.0034	0.0028	0.0028
	AAS	--	--	--	--	--	--
Be	ICP	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0011
	AAS	--	--	--	--		
Cd	ICP	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0011
	AAS	--	--	--	--	--	--
Cr	ICP	<0.0026	<0.0024	<0.0026	<0.0026	<0.0026	<0.0027
	AAS	--	--	--	--	--	--
Cu	ICP	0.0029	0.0025	<0.0010	0.0073	0.0068	0.0065
	AAS	--	--	--	--	--	--
	AAS	0.24	0.23	0.12	0.27	0.29	0.32
Pb	ICP	0.025	0.020	0.019	0.058	0.039	0.047
	AAS	<0.010	<0.010	<0.010	0.014	0.010	0.011
Mn	ICP	0.018	0.015	0.014	0.029	0.024	0.025
	AAS	0.020	0.019	0.018	0.027	0.026	0.026
Hg	ICP	--	--	--	--	--	--
	AAS	--	--	--	--	--	--
Ni	ICP	0.0110	0.011	0.010	0.018	0.016	0.0181
	AAS	0.059	0.051	0.059	0.047	0.063	0.078
Se	ICP	<0.021	<0.019	<0.020	<0.020	<0.020	<0.021
	AAS	<0.10	<0.098	<0.10	<0.10	<0.10	<0.11
Ag	ICP	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0011
	AAS	--	--	--	--	--	--
Tl	ICP	--	--	--	--	--	--
	AAS	--	--	--	--	--	--
Zn	ICP	<0.0010	<0.0010	<0.0010	0.0029	<0.0010	<0.0011
	AAS	--	--	--	--	--	--

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[a] Unless otherwise noted, concentrations are mg/L for Schemes 1 and 2.

[b] EP toxicity test with leachate digestion; aqueous phase only.

[c] Digestion according to EPA[2]. Sample stored at room temperature.

-- Not analyzed

APPENDIX F

RAW DATA FROM PHASE 2 ANALYSES OF LOW-pH SLUDGE

This appendix summarizes results of Phase 2 analyses of low-pH sludge. For all samples except the pH2 sample, the pH was adjusted to the appropriate initial pH (4, 6, or 8) with 1 M NaOH. Samples were then extracted at 0, 2, 8, and 16 hours following pH adjustment according to methods outlined by EPA (1980). Analyses were performed by ICP and AAS on undigested leachates. Results of Phase 2 analyses are presented in Table F-1.

Table F-1.

Element	Time Interval (hr)	Method	Concentration (mg/L) pH 2	pH 4	pH 6	pH 8
Al	0	ICP	14.7;14.7;14.5 (14.6 ± 0.1)	12.8;13.7;13.7 (13.4 ± 0.5)	2.63;2.36;2.83 (2.61 ± 0.23)	1.14;1.06;1.39 (1.20 ± 0.17)
		AAS	13.9;14.7;14.5 (14.4 ± 0.4)	11.5;12.4;12.5 (12.1 ± 0.6)	2.19;1.88;2.29 (2.12 ± 2.21)	1.16;1.06;1.26 (1.16 ± 0.10)
	2	ICP		11.5; 11.2; 12.0 (11.6 ± 0.4)	2.23; 3.24; 2.47 (2.65 ± 0.53)	1.37; 1.00; 1.36 (1.24 ± 0.21)
		AAS		12.1; 11.9; 12.1 (12.0 ± 0.1)	1.98; 2.60; 2.40 (2.33 ± 0.32)	1.78; 1.57; 1.88 (1.74 ± 0.16)
	8	ICP		13.3; 11.7; 11.8 (12.3 ± 0.9)	<1.25;<1.25;<1.25 (<1.25)	<1.25;<1.25;<1.25 (<1.25)
		AAS		13.2; 12.6; 12.6 (12.8 ± 0.3)	<0.50;<0.50;0.54 (<0.050)	0.95; 1.06; 0.75 (0.92 ± 0.16)
	16	ICP		12.9; 13.5; 13.2 (13.2 ± 0.3)	<1.25;<1.25;<1.25 (<1.25)	<1.25;<1.25;<1.25 (<1.25)
		AAS		11.5; 12.8; 12.5 (12.3 ± 0.7)	<0.50; 0.54;<0.050 (<0.050)	0.64;<0.050;<0.050 (<0.050)
Sb	0	ICP	<0.20;<0.20;<0.20 (<0.20)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.20;<0.20;<0.20 (<0.20)	<0.20;<0.20;<0.20 (<0.20)	<0.20;<0.20;<0.20 (<0.20)	<0.20;<0.20;<0.20 (<0.20)
	2	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		<0.20;<0.20;<0.20 (<0.20)	<0.20;<0.20;<0.20 (<0.20)	<0.20;<0.20;<0.20 (<0.20)
	8	ICP		<5;<5;<5	<5;<5;<5	<5;<5;<5

Table F-1. (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
As	0	ICP	0.520;0.468;0.422 (0.470 + 0.049)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	0.098;0.089;0.089 (0.092 ± 0.005	0.079;0.098;0.079 (0.085 ± 0.011)	0.089;0.098;0.089 (0.092 ± 0.005)	0.089;0.089;0.098 (0.092 ± 0.005)
	2	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		0.089;0.089;0.079 (0.086 ± 0.006)	0.089;0.079;0.079 (0.082 ± 0.006)	0.079;0.079;0.079 (0.079 ± 0.000)
	8	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		0.089;0.079;0.079 (0.082 ± 0.006)	0.089;0.089;0.089 (0.089 ± 0.000)	0.079;0.079;0.079 (0.079 ± 0.000)
	16	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		0.089;0.089;0.089 (0.089 ± 0.000)	0.089;0.098;0.079 (0.089 ± 0.010)	0.089;0.089;0.089 (0.089 ± 0.000)
Ba	0	ICP	0.145;0.109;0.123 (0.126 ± 0.018)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS	<0.50;0.51;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	2	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	8	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	16	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)

Table F-1. (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Be	0	ICP	<0.010;<0.010; <0.010 (<0.010)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS	<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)
	2	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)
	8	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)
	16	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)	<0.010;<0.010; < 0.010 (<0.010)
Cd	0	ICP	0.28;0.33;0.23 (0.28 ± 0.05)	<0.25;<0.25;0.33 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS	0.23;0.23;0.22 (0.23 ± 0.01)	0.20;0.20;0.20 (0.20 ± 0.00)	0.094;0.096;0.092 (0.094 ± 0.002)	0.072;0.072;0.074 (0.073 ± 0.001)
	2	ICP		<0.25;<0.25;0.26 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		0.21;0.20;0.21 (0.21 ± 0.01)	0.092;0.096;0.094 (0.094 ± 0.002)	0.074;0.070;0.070 (0.071 ± 0.002)
	8	ICP		<0.25;<0.25;0.26 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		0.20;0.20;0.21 (0.20 ± 0.01)	0.088;0.085;0.085 (0.086 ± 0.002)	0.070;0.068;0.066 (0.068 ± 0.002)
	16	ICP		0.25;0.30;0.28 (0.28 ± 0.02)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		0.20;0.21;0.21 (0.21 ± 0.01)	0.088;0.085;0.083 (0.085 ± 0.003)	0.068;0.068;0.066 (0.067 ± 0.001)

Table F-1. (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Cr	0	ICP	14.9;14.8;14.5 (14.7 ± 0.2)	0.84;1.03;0.80 (0.89 ± 0.12)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS	16.7;17.3;16.0 (16.7 ± 0.7)	0.99;1.20;1.09 (1.09 ± 0.11)	<0.10;<0.10;<0.10 (<0.10)	<0.10;<0.10;<0.10 (<0.10)
	2	ICP		0.57;0.61;0.68 (0.62 ± 0.06)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		0.84;0.79;0.88 (0.84 ± 0.05)	<0.10;<0.10;<0.10 (<0.10)	<0.10;<0.10;<0.10 (<0.10)
	8	ICP		2.45;0.55;0.57 (1.19 ± 1.09)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		2.66;0.77;0.78 (1.40 ± 1.09)	<0.10;<0.10;<0.10 (<0.10)	<0.10;<0.10;<0.10 (<0.10)
	16	ICP		0.75;1.63;0.76 (1.05 ± 0.51)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		0.95;1.78;0.95 (1.23 ± 0.48)	<0.10;<0.10;<0.10 (<0.10)	<0.10;<0.10;<0.10 (<0.10)
Cu	0	ICP	2.68;2.81;2.35 (2.61 ± 0.24)	1.77;1.89;1.88 (1.85 ± 0.07)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS	2.09;2.30;2.00 (2.13 ± 0.15)	1.51;1.64;1.62 (1.59 ± 0.07)	0.15;0.11;0.17 (0.14 ± 0.03)	0.093;0.093;0.093 (0.093 ± 0.000)
	2	ICP		1.74;1.67;1.70 (1.70 ± 0.04)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		1.62;1.60;1.62 (1.62 ± 0.01)	0.17;0.15;0.19 (0.17 ± 0.02)	0.093;0.074;0.074 (0.080 ± 0.011)
	8	ICP		1.73;1.67;1.81 (1.74 ± 0.07)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		1.62;1.58;1.64 (1.61 ± 0.03	0.11;0.13;0.15 (0.13 ± 0.02)	<0.050;<0.050; <0.050 (<0.050)
	16	ICP		1.82;1.79;1.86 (1.82 ± 0.04)	<0.63;<0.63;<0.63 (<0.63)	<0.63;<0.63;<0.63 (<0.63)
		AAS		1.60;1.64;1.62 (1.62 ± 0.02)	0.11;0.13;0.15 (0.13 ± 0.02)	<0.050;<0.050; <0.050 (<0.050)

Table F-1. (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Fe	0	ICP	1610;1610;1550 (1590 ± 30)	1150;1480;1450 (1360 ± 180)	231;206;204 (213 ± 15)	6.32;4.18;3.91 (4.80 ± 1.32)
		AAS	1590;1610;1530 (1580 ± 40)	1210;1310;1330 (1280 ± 60)	204;185;188 (192 ± 10)	6.59;4.44;4.75 (5.26 ± 1.16)
	2	ICP		1330;1260;1300 (1300 ± 30)	227;244;192 (221 ± 27)	8.71;5.59;6.04 (6.78 ± 1.69)
		AAS		1330;1310;1320 (1320 ± 10)	185;204;161 (183 ± 22)	9.96;5.97;6.59 (7.51 ± 2.15)
	8	ICP		1320;1290;1350 (1320 ± 30)	172;183;181 (179 ± 6)	4.88;9.99;1.75 (5.54 ± 4.16)
		AAS		1340; 1300;1370 (1340 ± 40)	158;161;158 (159 ± 2)	5.67;10.6;1.99 (6.09 ± 4.32)
	16	ICP		1490;1510;1510 (1500 ± 10)	140;150;145 (145 ± 5)	2.57;4.33;4.97 (3.96 ± 1.24)
		AAS		1260;1300;1300 (1290 ± 20)	130;136;133 (133 ± 3)	3.22;3.52;5.36 (4.03 ± 1.16)
Pb	0	ICP	4.31;4.85;4.63 (4.60 ± 0.27)	5.27;4.95;4.23 (4.82 ± 0.53)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	4.05;4.12;3.96 (4.04 ± 0.08)	2.46;2.59;2.43 (2.49 ± 0.09)	0.15;0.12;0.15 (0.14 ± 0.02)	<0.10;<0.10; <0.10 (<0.10)
	2	ICP		2.17;2.63;3.43 (2.74 ± 0.64)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		2.59;2.59;2.78 (2.65 ± 0.11)	0.15;0.18;0.18 (0.17 ± 0.02)	<0.10;<0.10; <0.10 (<0.10)
	8	ICP		4.23;3.27;3.69 (3.73 ± 0.48)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		3.22;2.71;2.94 (2.96 ± 0.26)	0.15;0.18;0.21 (0.18 ± 0.03)	<0.10;<0.10; <0.10 (<0.10)
	16	ICP		3.16;3.72;4.06 (3.65 ± 0.45)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		2.87;3.54;2.81 (3.07 ± 0.41)	0.15;0.15;0.12 (0.14 ± 0.02)	<0.10;<0.10; <0.10 (<0.10)

Table F-1. (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Mn	0	ICP	30.0;30.0;28.9 (29.6 ± 0.6)	27.3;28.9;28.2 (28.1 ± 0.8)	12.5;12.5;12.3 (12.4 ± 0.1)	9.68;9.35;9.49 (9.51 ± 0.17)
		AAS	30.4;31.0;29.5 (30.3 ± 0.8)	26.5;28.0;27.4 (27.3 ± 0.8)	12.7;12.7;12.7 (12.7 ± 0.0)	9.95;9.65;9.35 (9.65 ± 0.30)
	2	ICP		26.6;25.3;26.6 (26.2 ± 0.8)	12.4;12.8;12.1 (12.4 ± 0.4)	9.09;8.57;8.79 (8.82 ± 0.26)
		AAS		27.4;26.5;27.4 (27.1 ± 0.5)	12.4;12.7;12.1 (12.4 ± 0.3)	9.35;9.05;9.35 (9.25 ± 0.17)
	8	ICP		26.8;26.5;28.1 (27.1 ± 0.9)	12.2;12.3;12.5 (12.3 ± 0.2)	8.82;8.76;8.48 (8.69 ± 0.18)
		AAS		27.1;26.8;28.0 (27.3 ± 0.6)	12.7;12.4;12.7 (12.6 ± 0.2)	9.05;9.05;9.05 (9.05 ± 0.00)
	16	ICP		27.9;27.2;27.8 (27.6 ± 0.4)	11.5;11.5;12.0 (11.7 ± 0.3)	8.95;9.02;8.75 (8.91 ± 0.14)
		AAS		27.4;26.8;27.7 (27.3 ± 0.5)	12.4;11.8;12.4 (12.2 ± 0.3)	9.35;9.05;9.05 (9.15 ± 0.17)
Hg	0	ICP	--	--	--	--
		AAS	<0.0001;0.0002; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)
	2	ICP		--	--	--
		AAS		<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)
	8	ICP		--	--	--
		AAS		<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)
	16	ICP		--	--	--
		AAS		<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)	<0.0001;<0.0001; <0.0001 (<0.0001)

Table F-1 (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Ni	0	ICP	75.4;75.4;72.4 (74.4±1.7)	70.8;75.3;73.0 (73.0±2.3)	29.5;29.5;28.8 (29.3±0.4)	21.4;20.7;20.7 (20.9±0.4)
		AAS	75.4;75.4;73.5 (74.8±1.1)	64.0;68.3;66.4 (66.2±2.2)	29.7;28.8;28.8 (29.1±0.5)	20.7;19.7;19.7 (20.0±0.6)
	2	ICP		67.3;63.3;67.1 (65.9±2.3)	28.8;28.7;27.9 (28.5±0.5)	19.6;18.8;18.8 (19.1±0.5)
		AAS		67.8;65.9;69.3 (67.7±1.7)	26.9;26.9;26.4 (26.7±0.3)	20.2;18.8;19.3 (19.4±0.7)
	8	ICP		67.8;66.4;71.0 (68.4±2.4)	12.0;13.5;13.4 (13.0±0.8)	15.2;16.2;16.6 (16.0±0.7)
		AAS		68.3;66.9;69.7 (68.3±1.4)	11.2;12.6;12.6 (12.1±0.8)	15.4;15.9;15.9 (15.7±0.3)
	16	ICP		73.7;71.3;72.6 (72.5±1.2)	11.1;13.5;12.7 (12.4±1.2)	18.9;16.0;16.3 (17.1±1.6)
		AAS		68.8;67.4;68.8 (68.3±0.8)	11.2;14.0;13.5 (12.9±1.5)	18.8;16.4;16.4 (17.2±1.4)
Se	0	ICP	<11;<11;<11 (<11)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.50;<0.50;<0.50	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	2	ICP	<11;<11;<11 (<11)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.50;<0.50;<0.50	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	8	ICP	<11;<11;<11 (<11)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.50;<0.50;<0.50	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)
	16	ICP	<11;<11;<11 (<11)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.50;<0.50;<0.50	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)	<0.50;<0.50;<0.50 (<0.50)

Table F-1 (continued)

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Ag	0	ICP	<0.010;<0.010; <0.010 (<0.010)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)
	2	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)
	8	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)
	16	ICP		<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)	<0.25;<0.25;<0.25 (<0.25)
		AAS		<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)	<0.010;<0.010; <0.010 (<0.010)
Tl	0	ICP	<1;<1;<1 (<1)	<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)
	2	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)
	8	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)
	16	ICP		<5;<5;<5 (<5)	<5;<5;<5 (<5)	<5;<5;<5 (<5)
		AAS		<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)	<0.20;<0.20<0.20 (<0.20)

Table F-1.

Element	Time Interval (hr)	Method	pH 2	pH 4	pH 6	pH 8
Zn	0	ICP	3530;3540;3310 (3460 + 130)	3210;3370;3350 (3310 + 90)	1400;1370;1380 (1380 + 20)	1110;1100;1120 (1110 + 10)
		AAS	3180;3$\bar{2}$00;3030 (3140 ± 90)	2810;2$\bar{9}$00;2790 (2830 ± 60)	1210;1$\bar{1}$90;1180 (1190 ± 20)	986;97$\bar{7}$;977 (980 ± 5)
	2	ICP		3180;3170;3240 (3200 + 40)	1350;1200;1410 (1320 + 110)	1080;1030;1060 (1060 + 30)
		AAS		2970;2$\bar{9}$20;3010 (2970 ± 50)	1140;1$\bar{1}$50;1290 (1190 ± 30)	961;90$\bar{3}$;936 (933 ± 29)
	8	ICP		3400;3310;3530 (3410 + 110)	811;759;805 (792 + 28)	858;926;894 (893 + 34)
		AAS		2970;2$\bar{8}$60;3070 (2970 ± 110)	755;7$\bar{1}$3;765 (744 ± 28)	762;8$\bar{3}$6;795 (798 ± 37)
	16	ICP		3380;3400;3390 (3390 + 10)	825;880;940 (882 + 58)	975;893;919 (929 + 42)
		AAS		2920;2$\bar{9}$60;2970 (2950 ± 30)	745;8$\bar{4}$8;848 (814 ± 59)	861;8$\bar{2}$8;836 (842 ± 17)

APPENDIX G

RAW DATA FROM PHASE 3 ANALYSES OF RIVER SEDIMENT

This appendix summarizes results of Phase 3 analyses of a standardized river sediment.

The sample of river sediment was dried, ground, and homogenized prior to use for the Phase 3 extractions and bulk analysis. Bulk concentrations of the elements of interest were determined by ERCO and verified by an outside laboratory.

Each sample was prepared for the EP extractions according to methods outlined by EPA (1980). After addition of distilled water to the dried sediment, the samples were spiked in triplicate at three different concentration levels representing 150%, 100%, and 50% of the total metal concentrations determined from the bulk analyses. An unspiked sample was also prepared and extracted with the spiked sediments. For each spike level a "blank spike" was prepared by adding the spikes to distilled water. This solution was utilized to verify the integrity of the sample spikes.

Raw data for the low pH extracts are presented in Table G-1. Raw data for the pH-5 extracts are presented in Table G-2. For each element presented in Tables G-1 and G-2, individual determinations performed by AAS and ICP are presented, along with the mean and standard deviation calculated from the triplicate determinations. A combined mean and standard deviation from the AAS and ICP data is also presented. Recoveries of metal spikes from the 50%, 100% and 150% "blank spikes" for the river sediment are presented in Tables G-3 through G-6.

TABLE G-1. SUMMARY OF PHASE 3 ANALYSES OF STANDARDIZED RIVER SEDIMENT (LOW pH)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Low pH Extract (ppm)[d]
Al	AAS	49.0; 52.0; 54.0 (51.7 ± 2.5)	42.5	122 ± 6	28.0; 27.0; 28.0 (27.7 ± 0.6)	28.75	96 ± 2	52; 52; 52 (52 ± 0)	14.5	359 ± 0	53.0
	ICP	30.4; 31.4; 28.4 (30.0 ± 1.5)	42.5	71 ± 4	11.4; 11.4; 11.4 (11.4 ± 0)	28.75	40 ± 0	41.6; 41.5; 41.5 (41.5 ± 0.04)	14.5	286 ± 0	53.6
	Comb.	40.9 ± 12.0	42.5	96 ± 28	19.5 ± 8.9	28.75	68 ± 31	46.8 ± 5.7	14.5	323 ± 39	--
Sb	AAS	2.2; 1.9; 1.9 (2.0 ± 0.2)	15	13 ± 1	1.2; 1.4; 1.4 (1.3 ± 0.1)	10	13 ± 1	<0.5; <0.5; 0.7	5.0	<10	<0.5
	ICP	2.5; 2.4; 2.4 (2.4 ± 0.1)	15	16 ± 1	1.3; 1.3; 1.4 (1.3 ± 0.1)	10	13 ± 1	0.55; 0.53; 0.54 (0.54 ± 0.01)	5.0	11 ± 0.2	<0.07
	Comb.	2.2 ± 0.26	15	15 ± 1.7	1.3 ± 0.08	10	13 ± 0.8	0.54 ± 0.01	5.0		—
As	AAS	0.033; 0.106; 0.050 (0.063 ± 0.038)	0.387	16 ± 10	0.112; 0.099; 0.122 (0.111 ± 0.011)	0.25	44 ± 4	0.023; 0.040; 0.036 (0.033 ± 0.009)	0.125	26 ± 7	0.059
	ICP	0.079; 0.089; 0.118 (0.095 ± 0.020)	0.387	25 ± 5	0.139; 0.212; 0.157 (0.169 ± 0.038)	0.25	68 ± 15	0.090; 0.060; 0.046 (0.065 ± 0.022)	0.125	52 ± 18	0.21
	Comb.	0.079 ± 0.033	0.387	20 ± 9	0.140 ± 0.041	0.25	56 ± 16	0.049 ± 0.023	0.125	39 ± 18	--
Ba	AAS	3.27; 3.17; 3.17 (3.20 ± 0.06)	3.5	91 ± 2	2.24; 2.33; 2.24 (2.27 ± 0.05)	2.3	99 ± 2	1.02; 1.11; 1.02 (1.05 ± 0.05)	1.15	91 ± 4	1.3
	ICP	3.38; 3.59; 3.45 (3.47 ± 011)	3.5	99 ± 3	2.23; 2.29; 2.17 (2.23 ± 0.06)	2.3	97 ± 3	0.99; 1.00; 1.00 (1.00 ± 0.006)	1.15	87 ± 0.5	1.0
	Comb.	3.34 ± 0.17	3.5	95 ± 5	2.25 ± 0.06	2.3	98 ± 3	1.02 ± 0.04	1.15	89 ± 4	--
Be	AAS	<0.025;<0.025;<0.025	0.037	<68	<0.025;<0.025;<0.025	0.025	NC	<0.025;<0.025;<0.025	0.0125	NC	<0.025
	ICP	0.037; 0.040; 0.039 (0.039 ± 0.002)	0.037	105 ± 5	0.025; 0.025; 0.025 (0.025 ± 0)	0.025	100 ± 0	0.010; 0.010; 0.010 (0.010 ± 0)	0.0125	80 ± 0	0.011
	Comb.	0.039 ± 0.002	0.037	105 ± 5	0.025 ± 0	0.025	100 ± 0	0.010 ± 0	0.0125	80 ± 0	—
Cd	AAS	0.039; 0.045; 0.039 (0.041 ± 0.003)	0.050	82 ± 6	0.024; 0.027; 0.024 (0.025 ± 0.002)	0.030	83 ± 7	0.021; 0.026; 0.041 (0.029 ± 0.010)	0.015	193 ± 67	0.026
	ICP	0.058; 0.061; 0.065 (0.061 ± 0.004)	0.050	122 ± 8	0.044; 0.040; 0.041 (0.042 ± 0.002)	0.030	140 ± 7	0.027; 0.027; 0.025 (0.026 ± 0.001)	0.015	173 ± 7	0.027
	Comb.	0.051 ± 0.012	0.050	102 ± 24	0.033 ± 0.009	0.030	110 ± 30	0.028 ± 0.007	0.015	187 ± 47	--
Cr	AAS	3.13; 3.13; 3.13 (3.13 ± 0)	3.0	104 ± 0	2.03; 1.93; 2.03 (2.00 ± 0.006)	2.0	100 ± 3	0.83; 0.83; 0.83 (0.83 ± 0)	1.0	83 ± 0	0.37
	ICP	3.10; 3.16; 3.10 (3.12 ± 0.04)	3.0	104 ± 1	2.02; 2.02; 2.01 (2.02 ± 0.01)	2.0	101 ± 0.5	0.87; 0.87; 0.87 (0.87 ± 0)	1.0	87 ± 0	0.33
	Comb.	3.13 ± 0.02	3.0	104 ± 1	2.01 ± 0.04	2.0	101 ± 2	0.85 ± 0.02	1.0	85 ± 2	--
Cu	AAS	2.27; 2.37; 2.27 (2.30 ± 0.06)	2.25	102 ± 3	1.67; 1.67; 1.57 (1.61 ± 0.10)	1.5	107 ± 7	0.77; 0.77; 0.77 (0.77 ± 0)	0.75	103 ± 0	0.83
	ICP	2.64; 2.73; 2.64 (2.67 ± 0.05)	2.25	119 ± 2	1.86; 1.88; 1.86 (1.87 ± 0.01)	1.5	125 ± 1	0.90; 0.91; 0.90 (0.90 ± 0.01)	0.75	120 ± 1	0.90
	Comb.	2.49 ± 0.21	2.25	111 ± 9	1.75 ± 0.13	1.5	117 ± 9	0.84 ± 0.07	0.75	112 ± 9	--

[a] Final pH = 1.40; 1.49; 1.39. [b] Final pH = 1.65; 1.66; 1.65. [c] Final pH = 3.28; 3.27; 3.26. [d] Final pH = 1.39.

Table G-1. Continued.

Element	Method	Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			Concentration in Low pH Extract (ppm)[d]
		Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	
Fe	AAS	85; 84; 84 (84 ± 0.6)	68.5	123 ± 1	56; 60; 56 (57 ± 2.3)	46	124 ± 5	30; 28; 28 (28.7 ± 1.2)	23	125 ± 5	46
	ICP	99; 100; 97 (99 ± 1.5)	68.5	145 ± 2	68; 68; 69 (69 ± 0.6)	46	150 ± 1	35; 34; 35 (34.7 ± 0.6)	23	151 ± 3	55
	Comb.	92 ± 8	68.5	134 ± 12	63 ± 6	46	137 ± 13	32 ± 3	23	139 ± 13	—
Pb	AAS	53; 53; 53 (53 ± 0)	60	88 ± 0	32; 35; 35 (34 ± 1.7)	40.5	84 ± 4	10; 10; 10 (10 ± 0)	20	50 ± 0	27
	ICP	62; 66; 66 (65 ± 2.3)	60	108 ± 4	42; 42; 43 (42 ± 0.6)	40.5	104 ± 1.5	15; 15; 14 (14.7 ± 0.6)	20	74 ± 3	33
	Comb.	59 ± 6.6	60	98 ± 11	38 ± 4.7	40.5	94 ± 12	12.3 ± 2.6	20	62 ± 13	—
Mn	AAS	11.8; 11.8; 11.8 (11.8 ± 0)	12.5	94 ± 0	7.8; 7.8; 7.8 (7.8 ± 0)	8.5	92 ± 0	5.3; 5.9; 4.8 (5.3 ± 0.5)	4.25	125 ± 12	5.2
	ICP	12.6; 13.0; 12.7 (12.8 ± 0.2)	12.5	102 ± 2	8.6; 8.6; 8.6 (8.6 ± 0)	8.5	101 ± 0	5.2; 5.2; 5.2 (5.2 ± 0)	4.25	122 ± 0	5.3
	Comb.	12.3 ± 0.5	12.5	98 ± 4	8.2 ± 0.4	8.5	96 ± 5	5.3 ± 0.4	4.25	125 ± 9	—
Hg	AAS	0.0010; 0.0011; 0.0012 (0.0011 ± 0.0001)	0.0020	55 ± 5	0.00092; 0.00084; 0.00093 (0.00089 ± 0.00004)	0.0010	89 ± 4	0.00075; 0.00075; 0.00057 (0.00069 ± 0.00010)	0.0005	138 ± 20	0.032
	ICP	<10	0.0020	NC	<10	0.0010	NC	<10	0.0005	NC	<10
	Comb.	0.0011 ± 0.0001	0.0020	55 ± 5	0.00089 ± 0.00004	0.0010	89 ± 4	0.00069 ± 0.00010	0.0005	138 ± 20	—
Ni	AAS	0.93; 0.91; 0.96 (0.93 ± 0.025)	0.625	149 ± 4	0.84; 0.79; 0.84 (0.82 ± 0.028)	0.425	193 ± 6.6	0.64; 0.62; 0.57 (0.61 ± 0.036)	0.225	271 ± 16	<0.1
	ICP	0.72; 0.71; 0.70 (0.71 ± 0.01)	0.625	114 ± 2	0.49; 0.52; 0.49 (0.50 ± 0.02)	0.425	118 ± 5	0.247; 0.254; 0.243 (0.248 ± 0.006)	0.225	110 ± 3	0.057
	Comb.	0.82 ± 0.12	0.625	131 ± 19	0.66 ± 0.18	0.425	155 ± 42	0.43 ± 0.20	0.225	191 ± 89	—
Se	AAS	0.018; 0.018; 0.018 (0.018 ± 0)	0.20	9 ± 0	<0.01	0.125	<8	<0.01	0.075	<13	0.013
	ICP	<0.1	0.20	<50	<0.1	0.125	NC	<0.1	0.075	NC	<0.1
	Comb.	0.018 ± 0	0.20	9 ± 0	<0.01	0.125	<8	<0.01	0.075	<13	—
Ag	AAS	0.04; 0.04; 0.04 (0.04 ± 0)	3.0	1.3 ± 0	0.045; 0.025; 0.025 (0.032 ± 0.011)	2.0	1.6 ± 0.6	0.016; 0.017; <0.015 (0.016 ± 0.001)	1.0	1.6 ± 0.1	<0.015
	ICP	0.038; 0.035; 0.040 (0.038 ± 0.003)	3.0	1.3 ± 0.1	0.026; 0.027; 0.032 (0.028 ± 0.003)	2.0	1.4 ± 0.2	0.019; 0.021; 0.017 (0.019 ± 0.002)	1.0	1.9 ± 0.2	<0.005
	Comb.	0.039 ± 0.002	3.0	1.3 ± 0.1	0.030 ± 0.008	2.0	1.5 ± 0.4	0.018 ± 0.002	1.0	1.8 ± 0.2	—
Tl	AAS	2.20; 1.90; 1.90 (2.0 ± 0.17)	2.25	89 ± 8	1.10; 1.20; 1.20 (1.16 ± 0.08)	1.5	77 ± 5	0.49; 0.41; 0.49 (0.46 ± 0.04)	0.75	61 ± 5	<0.25
	ICP	1.93; 1.90; 1.89 (1.90 ± 0.2)	2.25	84 ± 9	1.10; 1.20; 1.20 (1.16 ± 0.08)	1.5	77 ± 5	0.54; 0.55; 0.48 (0.52 ± 0.04)	0.75	69 ± 5	<0.1
	Comb.	1.95 ± 0.12	2.25	87 ± 5	1.17 ± 0.05	1.5	78 ± 3	0.49 ± 0.05	0.75	65 ± 7	—
Sn	AAS	6.5; 6.5; 6.5 (6.5 ± 0)	7.0	93 ± 0	4.0; 4.3; 3.9 (4.1 ± 0.2)	4.5	91 ± 4	1.6; 1.8; 1.7 (1.7 ± 0.1)	2.25	76 ± 4	3.4
	ICP	6.7; 6.9; 6.8 (6.8 ± 0.1)	7.0	97 ± 1	4.5; 4.5; 4.3 (4.4 ± 0.1)	4.5	98 ± 2	1.8; 1.8; 1.8 (1.8 ± 0)	2.25	80 ± 0	3.4
	Comb.	6.7 ± 0.2	7.0	96 ± 3	4.3 ± 0.25	4.5	96 ± 6	1.75 ± 0.08	2.25	78 ± 4	—

[a] Final pH = 1.61; 1.59; 1.60.

[b] Final pH = 1.70; 1.60; 1.70.

[c] Final pH = 2.62; 2.55; 2.57.

[d] Final pH = 1.6.

TABLE G-2. SUMMARY OF PHASE I ANALYSES OF STANDARDIZED RIVER SEDIMENT (pH 5)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Al	AAS	2.52;0.97;0.97 (1.49±0.89)	42.5	3±2	0.97;1.09;0.86 (0.97±0.11)	28.75	3±1	0.53;0.64;0.64 (0.60±0.06)	14.5	4±0	<0.50
	ICP	1.82;0.63;0.80 (1.08±0.64)	42.5	2±2	0.95;0.83;0.65 (0.81±0.15)	28.75	3±1	0.34;0.42;0.37 (0.38±0.04)	14.5	3±1	0.18
	Comb.	1.29±0.73	42.5	3±2	0.89±0.15	28.75	3±1	0.49±0.13	14.5	3±1	
Sb	AAS	<0.20;<0.20; <0.20 (<0.20)	15	<1	<0.20;<0.20; <0.20 (<0.20)	10	<2	<0.20;<0.02; <0.20 (<0.20)	5	<4	<0.20
	ICP	<0.20;<0.20; <0.20 (<0.20)	15	<1	<0.20;<0.20; <0.20 (<0.20)	10	<2	<0.20;<0.21; <0.20 (<0.20)	5	<4	<0.20
	Comb.	<0.20	15	<1	<0.20	10	<2	<0.20	5	<4	
As	AAS	0.120;0.137; 0.128 (0.128±0.009)	0.385	33±3	0.082;0.086; 0.090 (0.086±0.004)	0.250	34±2	0.036;0.037; 0.037 (0.037±0.001)	0.125	30±1	<0.010
	ICP	<0.200;<0.200; <0.200 (<0.200)	0.385	<52	<0.200;<0.200; <0.200 (<0.200)	0.250	<80	<0.200;<0.200; <0.200 (<0.200)	0.125	NC	<0.200
	Comb.	<0.200	0.385	33±3	<0.200	0.250	34±2	<0.200	0.125	30±1	
Ba	AAS	3.63;3.85;3.92 (3.80±0.15)	3.5	108±5	2.62;2.69;2.76 (2.69±0.07)	2.25	120±4	1.38;1.46;1.17 (1.34±0.15)	1.15	117±14	<0.50
	ICP	3.32;3.29;3.32 (3.31±0.02)	3.5	95±1	2.27;.25;2.29 (2.27±0.02)	2.25	101±1	1.09;1.17;1.12 (1.13±0.04)	1.15	98±4	0.053
	Comb.	3.56±0.29	3.5	102±8	2.48±0.23	2.25	110±10	1.23±0.15	1.15	107±13	
Be	AAS	0.048;0.034; 0.034 (0.039±0.008)	0.375	10±2	0.028;0.032; 0.032 (0.031±0.002)	0.250	12±1	<0.010;<0.010; 0.013 (<0.010)	0.125	<8	<0.010
	ICP	0.043;0.032; 0.035 (0.037±0.006)	0.375	10±1	0.031;0.033; 0.034 (0.033±0.002)	0.250	13±1	<0.010;<0.010; <0.010 (<0.010)	0.125	<8	<0.010
	Comb.	0.038±0.006	0.375	10±2	0.032±0.002	0.250	13±1	<0.010	0.125	<8	
Cd	AAS	0.070;0.072; 0.085 (0.076±0.008)	0.050	151±16	0.048;0.048 0.048 (0.048±0.000)	0.030	160±0	0.030;0.033; 0.033 (0.032±0.002)	0.020	160±9	<0.010
	ICP	0.067;0.072; 0.082 (0.074±0.008)	0.050	147±15	0.052;0.052; 0.057 (0.054±0.003)	0.030	179±10	0.034;0.038; 0.035 (0.036±0.002)	0.020	178±10	<0.010
	Comb.	0.075±0.007	0.050	149±14	0.051±0.004	0.030	169±12	0.034±0.003	0.020	169±13	

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[a] Final pH = 5.0;5.1;5.1. [b] Final pH = 5.0;5.1;5.1. [c] Final pH = 5.0;5.1;5.1. [d] Final pH = 5.2.

NC = Not calculated.

Table G-2. (continued)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Cr	AAS	<0.10;<0.10; <0.10 (<0.10)	3.0	<3	<0.10;<0.10; <0.10 (<0.10)	2.0	<5	<0.10;<0.10; <0.10 (<0.10)	1.0	<10	<0.10
	ICP	<0.025;<0.025 <0.025 (<0.025)	3.0	<1	<0.025;<0.025 <0.025 (<0.025)	2.0	<1	<0.025;<0.025 <0.025 (<0.025)	1.0	<3	<0.025
	Comb.	<0.025	3.0	<1	<0.025	2.0	<1	<0.025	1.0	<3	
Cu	AAS	0.49;0.39;0.47 (0.45±0.05)	2.25	20±3	0.19;0.21;0.22 (0.21±0.02)	1.50	14±1	<0.050;0.055; 0.055 (0.055±0.000)	0.75	7±0	<0.050
	ICP	0.52;0.41;0.51 (0.48±0.06)	2.25	21±3	0.24;0.26;0.26 (0.25±0.01)	1.50	17±1	0.076;0.084; 0.079 (0.080±0.004)	0.75	11±1	<0.025
	Comb.	0.47±0.05	2.25	21±3	0.23±0.03	1.50	15±2	0.067±0.15	0.75	8±4	
Fe	AAS	1.13;0.86;1.02 (1.00±0.14)	68.5	1±1	0.82;0.97;0.94 (0.91±0.08)	46	2±0	0.55;0.47;0.45 (0.49±0.05)	23	2±0	0.14
	ICP	1.06;0.85;1.03 (0.98±0.11)	68.5	2±1	0.84;1.02;1.00 (0.95±0.10)	46	2±0	0.55;0.51;0.48 (0.51±0.04)	23	2±0	0.078
	Comb.	0.99±0.11	68.5	2±1	0.93±0.08	46	2±0	0.50±0.04	23	2±0	
Pb	AAS	22.8;21.2;18.8 (20.9±2.0)	60	35±4	10.9;11.2;11.5 (11.2±0.3)	40.5	28±1	3.05;3.69;3.28 (3.34±0.32)	20	16±2	0.30
	ICP	21.9;19.1;19.3 (20.1±1.6)	60	34±3	11.2;11.4;11.7 (11.4±0.3)	40.5	28±1	3.01;3.74;3.34 (3.36±0.37)	20	17±2	0.28
	Comb.	20.5±1.7	60	34±3	11.3±0.3	40.5	28±1	3.35±0.31	20	17±2	
Mn	AAS	15.1;15.8;15.1 (15.3±0.4)	12.5	123±3	10.5;10.8;10.5 (10.6±0.2)	8.5	125±2	5.78;6.09;6.09 (5.99±0.18)	4.25	141±4	1.23
	ICP	13.1;13.6;13.7 (13.5±0.3)	12.5	108±3	10.2;9.9;9.9 (10.0±0.2)	8.5	117±2	5.59;5.93;5.68 (5.73±0.18)	4.25	135±4	1.25
	Comb.	14.4±1.1	12.5	115±8	10.3±0.4	8.5	121±5	5.86±0.21	4.25	138±5	
Hg	AAS	<0.0001;<0.0001 <0.0001 (<0.0001)	0.0020	<5	<0.0001;<0.0001; <0.0001 (<0.0001)	0.0010	<10	<0.0001;<0.0001; <0.0001 (<0.0001)	0.0005	<20	<0.0001
	ICP	--	0.0020	--	--	0.0010	--	--	0.0005	--	--
	Comb.	<0.0001	0.0020	<5	<0.0001	0.0010	<10	<0.0001	0.0005	<20	

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[a] Final pH = 5.0;5.1;5.1.
[b] Final pH = 5.0;5.1;5.1.
[c] Final pH = 5.0;5.1;5.1.
[d] Final pH = 5.2.

Table G-2. (continued)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Ni	AAS	0.60;0.58;0.58 (0.59±0.01)	0.625	94±2	0.38;0.38;0.38 (0.38±0.00)	0.425	89±0	0.15;0.18;0.18 (0.17±0.02)	0.225	76±8	<0.050
	ICP	0.57;0.58;0.59 (0.58±0.01)	0.625	93±2	0.41;0.42;0.43 (0.42±0.01)	0.425	99±3	0.21;0.25;0.23 (0.23±0.02)	0.225	102±9	<0.050
	Comb.	0.58±0.01	0.625	93±2	0.40±0.02	0.425	94±6	0.20±0.04	0.225	89±16	
Se	AAS	0.031;0.040; 0.031 (0.034±0.005)	0.200	17±2	0.027;0.026; 0.026 (0.026±0.001)	0.125	21±1	0.009;0.006; 0.006 (0.007±0.002)	0.075	9±2	<0.005
	ICP	<0.200;<0.200; <0.200 (<0.200)	0.200	NC	<0.200;<0.200; <0.200 (<0.200)	0.125	NC	<0.200;<0.200; <0.200 (<0.200)	0.075	NC	<0.200
	Comb.	0.034±0.005	0.200	17±2	0.026±0.001	0.125	21±1	0.007±0.002	0.075	9±2	
Ag	AAS	<0.010;<0.010 <0.010 (<0.010)	3.0	<1	<0.010;<0.010 <0.010 (<0.010)	2.0	<1	<0.010;<0.010 <0.010 (<0.010)	1.0	<1	<0.010
	ICP	<0.010;<0.010 <0.010 (<0.010)	3.0	<1	<0.010;<0.010 <0.010 (<0.010)	2.0	<1	<0.010;<0.010 <0.010 (<0.010)	1.0	<1	<0.010
	Comb.	<0.010	3.0	<1	<0.010	2.0	<1	<0.010	1.0	<1	
Tl	AAS	1.49;1.36;1.26 (1.37±0.12)	2.25	61±5	0.77;0.70;0.73 (0.73±0.04)	1.5	49±2	0.30;0.24;0.30 (0.28±0.03)	0.75	37±5	<0.20
	ICP	1.10;1.09;1.18 (1.12±0.05)	2.25	50±2	0.64;0.68;0.63 (0.65±0.03)	1.5	43±2	0.24;0.20;0.21 (0.22±0.02)	0.75	29±3	<0.25
	Comb.	1.24±0.16	2.25	55±7	0.69±0.05	1.5	46±3	0.25±0.04	0.75	33±6	
Zn	AAS	7.32;7.63;7.42 (7.46±0.16)	7.0	107±2	4.93;4.83;5.14 (4.97±0.16)	4.5	110±4	2.44;3.17;2.86 (2.82±0.37)	2.25	125±16	0.23
	ICP	6.75;6.99;7.16 (6.96±0.21)	7.0	99±3	4.69;4.73;4.85 (4.76±0.08)	4.5	106±2	2.34;2.48;2.28 (2.37±0.10)	2.25	105±5	0.23
	Comb.	7.21±0.31	7.0	103±5	4.86±0.16	4.5	108±4	2.59±0.34	2.25	115±15	

[a] Final pH = 5.0;5.1;5.1. [b] Final pH = 5.0;5.1;5.1. [c] Final pH = 5.0;5.1;5.1. [d] Final pH = 5.2.

NC = Not calculated.

TABLE G-3. SUMMARY OF RECOVERIES OF METALS FROM THE 50% "BLANK SPIKE" FOR RIVER SEDIMENT (pH 5)

Element	Concentration Added (ppm)	Recovery (%)
Al	14.5	94
Sb	5.00	98
As	0.125	107
Ba	1.15	95
Be	0.125	101
Cd	0.020	150
Cr	1.00	100
Cu	0.750	93
Fe	23.0	94
Pb	20.0	104
Mn	4.25	97
Hg	0.0005	300
Ni	0.225	67
Se	0.075	103
Ag	1.00	17
Tl	0.750	97
Zn	2.25	100

TABLE G-4. SUMMARY OF RECOVERIES OF METALS FROM THE 100% "BLANK SPIKE" FOR RIVER SEDIMENT (pH 5)

Element	Concentration Added (ppm)	Recovery (%)
Al	28.75	97
Sb	10.0	102
As	0.250	114
Ba	2.30	98
Be	0.250	95
Cd	0.030	130
Cr	2.00	101
Cu	1.50	95
Fe	46.0	100
Pb	40.5	108
Mn	8.50	101
Hg	0.0010	300
Ni	0.425	85
Se	0.125	108
Ag	2.00	9
Tl	1.50	101
Zn	4.50	103

TABLE G-5. SUMMARY OF RECOVERIES OF METALS FROM THE 100% "BLANK SPIKE" FOR RIVER SEDIMENT (LOW pH)

Element	Concentration Added (ppm)	Recovery (%)
Al	28.75	99
Sb	10	114
As	0.25	68
Ba	2.3	105
Be	0.025	112
Cd	0.030	73
Cr	2.0	106
Cu	1.5	105
Fe	46	101
Pb	40.5	112
Mn	8.5	101
Ni	0.425	109
Se	0.125	96
Ag	2.0	1.3
Tl	1.5	99
Zn	4.5	104

TABLE G-6. SUMMARY OF RECOVERIES OF METALS FROM THE 150% "BLANK SPIKE" FOR RIVER SEDIMENT (pH 5).

Element	Concentration Added (ppm)	Recovery (%)
Al	42.5	98
Sb	15.0	99
As	0.385	112
Ba	3.50	89
Be	0.375	74
Cd	0.050	96
Cr	3.00	102
Cu	2.25	97
Fe	68.5	98
Pb	60.0	105
Mn	12.5	103
Hg	0.0020	245
Ni	0.625	93
Se	0.200	113
Ag	3.00	5
Tl	2.25	101
Zn	7.00	99

APPENDIX H

RAW DATA FROM PHASE 3 ANALYSES OF FLY ASH

This appendix summarizes results of Phase 3 analyses of a standardized fly ash. The sample of fly ash was dried, ground, and homogenized prior to use for Phase 3 extraction and bulk analysis. Bulk concentrations of the elements of interest were determined by ERCO and verified by an outside laboratory.

Each sample was prepared for the EP extraction according to methods outlined by EPA[1]. After addition of distilled water to the dried ash, the samples were spiked in triplicate at these different concentration levels representing 150%, 100%, and 50% of the total metal concentrations determined from the bulk analysis. For each spike level, a "blank spike" was prepared by adding the spikes to distilled water. This solution was utilized to verify the integrity of the sample spikes.

Raw data for the low pH extracts are presented in Table H-1. Raw data for the pH-5 extracts are presented in Table H-2. For each element presented in Tables H-1 and H-2, individual determinations performed by AAS and ICP are presented, along with the mean and standard deviation calculated from the triplicate determinations. A combined mean and standard deviation from the AAS and ICP data is also presented. Recoveries of metal spikes from the 50%, 100% and 150% "blank spikes" for the fly ash are presented in Tables H-3 through H-6.

TABLE II-1. SUMMARY OF PHASE I ANALYSIS OF FLY ASH (LOW pH)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Low pH Extract (ppm)[d]
Al	AAS	131; 133; 127 (130 ± 3.1)	105	124 ± 2.7	90; 93; 90 (91 ± 1.7)	70	130 ± 2.4	32; 33; 34 (33 ± 1)	35	94 ± 5.5	66
	ICP	99; 100; 98 (99 ± 1)	105	94 ± 0.95	58; 61; 61 (60 ± 1.7)	70	86 ± 2.4	15; 15; 15 (15 ± 0)	35	43 ± 0	67
	Comb.	115 ± 17	105	110 ± 16	76 ± 17	70	109 ± 24	24 ± 10	35	69 ± 29	--
Sb	AAS	1.6; 1.6; 1.6 (1.6 ± 0)	10	16 ± 0	0.69; 0.93; 0.69 (0.77 ± 0.14)	5	15 ± 2.8	<0.5; <0.5; <0.5	2.5	<20	<0.5
	ICP	1.44; 1.50; 1.54 (1.49 ± 0.05)	10	15 ± 0.5	0.53; 0.96; 0.56 (0.68 ± 0.24)	5	14 ± 4.8	0.29; 0.23; 0.25 (0.26 ± 0.03)	2.5	10 ± 1.2	0.04
	Comb.	1.38 ± 0.39	10	14 ± 4	0.73 ± 0.18	5	15 ± 4	0.26 ± 0.03	2.5	10 ± 1.2	--
As	AAS	1.55; 0.85; 2.55 (1.65 ± 0.85)	2.6	63 ± 32	1.10; 2.10; 1.2 (1.47 ± 0.55)	1.75	84 ± 31	0.093; 0.065; 0.111 (0.090 ± 0.023)	0.90	10 ± 2.6	1.45
	ICP	2.17; 2.40; 2.17 (2.25 ± 0.13)	2.6	87 ± 5	1.81; 2.45; 2.02 (2.09 ± 0.33)	1.75	119 ± 19	0.099; 0.117; 0.240 (0.152 ± 0.076)	0.90	17 ± 8.4	1.0
	Comb.	1.95 ± 0.64	2.6	75 ± 25	1.78 ± 0.53	1.75	102 ± 30	0.121 ± 0.06	0.90	13 ± 7	--
Ba	AAS	2.0; 1.9; 1.9 (1.93 ± 0.06)	14.25	13.5 ± 0.42	1.8; 1.8; 1.8 (1.8 ± 0)	9.5	19 ± 0	1.3; 1.3; 1.3 (1.3 ± 0)	4.75	27 ± 0	1.2
	ICP	0.86; 1.09; 0.98 (0.98 ± 0.12)	14.25	6.9 ± 0.8	0.80; 0.49; 0.93 (0.74 ± 0.22)	9.5	7.8 ± 2.3	0.64; 0.67; 0.65 (0.65 ± 0.01)	4.75	14 ± 0.2	0.86
	Comb.	1.46 ± 0.53	14.25	10 ± 3.7	1.27 ± 0.60	9.5	13 ± 6	0.98 ± 0.35	4.75	21 ± 7	--
Be	AAS	0.23; 0.25; 0.24 (0.24 ± 0.01)	0.30	80 ± 3.3	0.16; 0.16; 0.15 (0.16 ± 0.01)	0.20	80 ± 5	0.080; 0.087; 0.097 (0.088 ± 0.009)	0.10	88 ± 9	<0.025
	ICP	0.31; 0.32; 0.31 (0.31 ± 0.01)	0.30	103 ± 3.3	0.20; 0.20; 0.21 (0.20 ± 0.01)	0.20	100 ± 5	0.095; 0.097; 0.093 (0.095 ± 0.002)	0.10	95 ± 2	0.031
	Comb.	0.28 ± 0.04	0.30	93 ± 13	0.18 ± 0.03	0.20	90 ± 15	0.092 ± 0.007	0.10	92 ± 7	--
Cd	AAS	1.5; 1.5; 1.5 (1.5 ± 0)	1.5	100 ± 0	0.96; 0.92; 0.99 (0.96 ± 0.03)	1.0	96 ± 3	0.46; 0.47; 0.45 (0.46 ± 0.01)	0.50	92 ± 2	0.012
	ICP	1.40; 1.43; 1.39 (141 ± 0.02	1.5	94 ± 1.3	1.04; 1.00; 1.08 (1.04 ± 0.04)	1.0	104 ± 4	0.54; 0.55; 0.52 (0.53 ± 0.02)	0.50	106 ± 4	<0.01
	Comb.	1.45 ± 0.05	1.5	97 ± 3.3	100 ± 0.06	1.0	100 ± 6	0.50 ± 0.04	0.50	100 ± 8	--
Cr	AAS	3.8; 3.9; 3.9 (3.9 ± 0.06)	3.6	108 ± 1.6	2.7; 2.7; 2.8 (2.7 ± 0.06)	2.5	108 ± 24	1.10; 1.00; 1.10 (1.07 ± 0.06)	1.25	86 ± 48	1.2
	ICP	3.6; 3.6; 3.6 (3.6 ± 0)	3.6	100 ± 0	2.5; 2.4; 2.5 (2.5 ± 0.06)	2.5	100 ± 2.4	0.89; 0.89; 0.88 (0.89 ± 0.01)	1.25	71 ± 0.8	1.2
	Comb.	3.7 ± 0.2	3.6	103 ± 5.6	2.6 ± 0.16	2.5	104 ± 6.4	0.98 ± 0.11	1.25	78 ± 8.8	--
Cu	AAS	2.6; 2.6; 2.5 (2.6 ± 0.06)	2.7	96 ± 2.2	1.7; 1.7; 1.7 (1.7 ± 0)	1.8	94 ± 0	0.81; 0.81; 0.81 (0.81 ± 0)	0.90	90 ± 0	0.69
	ICP	2.8; 2.8; 2.8 (2.8 ± 0)	2.7	104 ± 0	1.9; 1.8; 1.9 (1.9 ± 0.06)	1.8	106 ± 3.3	0.84; 0.87; 0.85 (0.85 ± 0.02)	0.90	94 ± 2.2	0.74
	Comb.	2.7 ± 0.13	2.7	100 ± 4.8	1.8 ± 0.10	1.8	100 ± 5.6	0.83 ± 0.03	0.90	92 ± 3.3	--

[a] Final pH = 1.61; 1.59; 1.60. [b] Final pH = 1.70; 1.60; 1.70. [c] Final pH = 2.62; 2.55; 2.57. [d] Final pH = 1.6.

TABLE B-1. Continued

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Low pH Extract (ppm)[d]
Fe	AAS	32; 23; 28 (28 ± 4.5)	36.5	77 ± 12	25; 32; 25 (27 ± 4)	24	113 ± 17	7.3; 6.0; 4.7 (6.0 ± 1.3)	12	50 ± 11	17
	ICP	35; 34; 34 (34 ± 0.6)	36.5	93 ± 1.6	34; 35; 35 (35 ± 1)	24	146 ± 4	10; 10; 10 (10 ± 0)	12	83 ± 0	10
	Comb.	31 ± 4.6	36.5	85 ± 13	31 ± 5	24	129 ± 21	8.0 ± 2.3	12	67 ± 19	--
Pb	AAS	<0.20; <0.20; <0.20	0.50	<40	<0.20; <0.20; <0.20	0.35	<57	<0.20; <0.20; <0.20	0.175	NC	<0.2
	ICP	<0.20; <0.20; <0.20	0.50	<40	<0.20; <0.20; <0.20	0.35	<57	<0.20; <0.20; <0.20	0.175	NC	<0.2
	Comb.	<0.2	0.50	<40	<0.20	0.35	<57	<0.20	0.175	NC	NC
Mn	AAS	2.2; 2.3; 2.3 (2.3 ± 0.06)	2.0	115 ± 3	1.3; 1.3; 1.3 (1.3 ± 0)	1.4	93 ± 0	0.70; 0.69; 0.69 (0.69 ± 0.01)	0.75	92 ± 1.3	0.26
	ICP	1.98; 2.00; 1.99 (2.0 ± 0.01)	2.0	100 ± 0.5	1.36; 1.35; 1.41 (1.4 ± 0.3)	1.4	100 ± 2	0.69; 0.69; 0.69 (0.69 ± 0)	0.75	92 ± 0	0.27
	Comb.	2.1 ± 0.16	2.0	105 ± 8	1.3 ± 0.05	1.4	93 ± 4	0.69 ± 0.01	0.75	92 ± 1	--
Hg	AAS	0.0027;0.0029;0.0013 (0.0023 ± 0.0009)	0.0020	115 ± 45	0.0015;0.0016;0.0017 (0.0016 ± 0.0001)	0.0010	160 ± 10	0.0007;0.0009;0.0007 (0.00077 ± 0.00012)	0.0005	154 ± 24	0.0005
	ICP	<10	0.0020	NC	<10	0.0010	NC	<10	0.0005	NC	--
	Comb.	0.0023 ± 0.0009	0.0020	115 ± 45	0.0016 ± 0.0001	0.0010	160 ± 10	0.00077 ± 0.00012	0.0005	154 ± 24	--
Ni	AAS	1.8; 1.9; 1.9 (1.9 ± 0.1)	1.4	136 ± 7	1.00; 1.10; 1.3 (1.1 ± 0.15)	0.90	122 ± 17	0.47; 1.07; 1.05 (0.86 ± 0.34)	0.50	172 ± 68	<0.1
	ICP	1.40; 1.46; 1.42 (1.43 ± 0.03)	1.4	100 ± 2	0.91; 0.91; 0.94 (0.92 ± 0.02)	0.90	102 ± 2	0.51; 0.49; 0.52 (0.51 ± 0.02)	0.50	102 ± 4	0.10
	Comb.	1.6 ± 0.2.	1.4	114 ± 14	1.03 ± 0.15	0.90	114 ± 17	0.69 ± 0.29	0.50	138 ± 58	--
Be	AAS	0.18; 0.35; 0.17 (0.23 ± 0.10)	0.255	90 ± 39	0.15; 0.25; 0.17 (0.19 ± 0.05)	0.20	95 ± 25	0.022; 0.022; 0.022 (0.022 ± 0)	0.10	22 ± 0	0.160
	ICP	<0.1; <0.1; <0.1	0.255	<40	<0.1; <0.1; <0.1	0.20	<50	<0.1; <0.1; <0.1	0.10	NC	<0.1
	Comb.	0.23 ± 0.10	0.255	90 ± 39	0.19 ± 0.05	0.20	95 ± 25	0.022 ± 0	0.10	22 ± 0	--
Ag	AAS	0.031; 0.034; 0.032 (0.032 ± 0.002)	3.0	1 ± 0.1	0.021; 0.021; 0.031 (0.024 ± 0.006)	2.0	1.2 ± 0.3	<0.015;<0.015;<0.015	1.0	<1.5	<0.015
	ICP	0.030; 0.030; 0.030 (0.030 ± 0)	3.0	1 ± 0	0.016; 0.014; 0.016 (0.015 ± 0.001)	2.0	0.75 ± 0.05	0.010; 0.010; 0.010 (0.010 ± 0)	1.0	1 ± 0	--
	Comb.	0.031 ± 0.002	3.0	1 ± 0.1	0.020 ± 006	2.0	1 ± 0.3	0.010 ± 0	1.0	1 ± 0	--
Tl	AAS	2.9; 2.9; 2.9 (2.9 ± 0)	3.0	97 ± 0	1.5; 1.4; 1.4 (1.4 ± 0.66)	1.5	93 ± 4	0.55; 0.47; 0.49 (0.50 ± 0.04)	0.75	67 ± 5	<0.25
	ICP	2.72; 2.67; 2.67 (2.69 ± 0.03)	3.0	90 ± 1	1.45; 1.38; 1.35 (1.39 ± 0.05)	1.5	93 ± 3	0.69; 0.67; 0.71 (0.69 ± 0.02)	0.75	92 ± 3	--
	Comb.	2.79 ± 0.12	3.0	93 ± 4	1.4 ± 0.05	1.5	93 ± 3	0.60 ± 0.11	0.75	80 ± 15	--
Zn	AAS	1.74; 2.24; 2.14 (2.04 ± 0.26)	2.25	91 ± 12	1.4; 1.3; 1.3 (1.3 ± 0.06)	1.5	89 ± 4	0.32; 0.32; 0.32 (0.32 ± 0)	0.75	43 ± 0	0.26
	ICP	2.24; 2.28; 2.24 (2.25 ± 0.02)	2.25	100 ± 1	1.52; 1.47; 1.56 (1.52 ± 0.05)	1.5	100 ± 3	0.34; 0.34; 0.34 (0.34 ± 0)	0.75	45 ± 0	0.27
	Comb.	2.15 ± 0.20	2.25	96 ± 9	1.43 ± 0.11	1.5	95 ± 7	0.33 ± 0.01	0.75	44 ± 1	--

[a] Final pH = 1.61; 1.59; 1.60. [b] Final pH = 1.70; 1.60; 1.70. [c] Final pH = 2.62; 2.55; 2.57. [d] Final pH = 1.6.

TABLE B-2. SUMMARY OF PHASE 3 ANALYSES OF FLY ASH (pH 5)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Al	AAS	85.5;86.6;84.5 (85.5±1.1)	105	81±1	58.7;55.6;56.7 (57.0±1.6)	70	81±3	11.3;16.4;18.5 (15.4±3.7)	35	44±11	3.43
	ICP	81.3;81.9;84.1 (82.4±1.5)	105	78±2	54.6;53.3;54.7 (54.2±0.8)	70	77±1	12.0;20.5;17.4 (16.6±4.3)	35	48±13	3.35
	Comb.	84.0±2	105	80±2	55.6±1.9	70	79±3	16.0±3.7	35	46±11	
Sb	AAS	3.14;3.26;3.11 (3.17±0.08)	10	32±1	0.64;0.91;0.91 (0.82±0.16)	5.0	16±3	<0.20;<0.20;<0.20 (<0.20)	2.5	<8	<0.20
	ICP	3.13;3.02;2.80 (2.98±0.17)	10	30±2	0.71;0.89;0.84 (0.81±0.09)	5.0	16±2	0.24;0.22;0.25 (0.24±0.02)	2.5	10±1	<0.20
	Comb.	3.08±0.16	10	31±2	0.82±0.11	5.0	16±2	0.24±0.02	2.5	10±1	
As	AAS	1.00;0.93;1.07 (1.00±0.07)	2.60	38±3	0.20;0.28;0.28 (0.25±0.05)	1.75	14±3	<0.010;<0.010;<0.010 (<0.010)	0.90	<1	0.035
	ICP	1.15;1.04;1.09 (1.09±0.06)	2.60	42±2	0.39;0.33;0.34 (0.35±0.03)	1.75	20±2	<0.20;<0.20;<0.20 (<0.20)	0.90	<22	<0.20
	Comb.	1.05±0.08	2.60	40±3	0.30±0.07	1.75	17±4	<0.010	0.90	<1	
Ba	AAS	1.09;1.17;1.02 (1.11±0.05)	14.25	8±1	0.51;0.51;<0.50 (0.51±0.01)	9.50	5±1	<0.50;<0.50;<0.50 (<0.50)	4.75	<10	<0.50
	ICP	0.96;0.92;0.88 (0.92±0.04)	14.25	6±1	0.51;0.42;0.43 (0.45±0.05)	9.50	5±1	0.36;0.48;0.40 (0.41±0.06)	4.75	9±1	<0.010
	Comb.	1.01±0.11	14.25	7±1	0.48±0.04	9.50	5±1	0.41±0.06	4.75	9±1	
Be	AAS	0.27;0.26;0.28 (0.27±0.01)	0.30	90±3	0.18;0.17;0.18 (0.18±0.01)	0.20	88±3	0.066;0.074;0.074 (0.071±0.004)	0.10	71±5	<0.010
	ICP	0.30;0.28;0.31 (0.030±0.02)	0.30	99±5	0.19;0.20;0.20 (0.20±0.01)	0.20	98±3	0.072;0.083;0.082 (0.079±0.006)	0.10	79±6	<0.010
	Comb.	0.28±0.02	0.30	94±6	0.19±0.02	0.20	93±6	0.075±0.006	0.10	75±6	
Cd	AAS	1.52;1.52;1.55 (1.53±0.02)	1.50	102±1	1.03;1.05;1.04 (1.04±0.01)	1.00	104±1	0.48;0.50;0.50 (0.49±0.01)	0.50	99±2	0.013
	ICP	1.53;1.51;1.54 (1.53±0.02)	1.50	102±1	102;103;1.04 (1.03±0.01)	1.00	103±1	0.51;0.52;0.52 (0.052±0.01)	0.50	103±1	<0.010
	Comb.	1.53±0.02	1.50	102±1	1.04±0.01	1.00	104±1	0.50±0.02	0.50	101±3	

[a] Final pH = 4.3;4.3;4.3. [b] Final pH = 4.2;4.2;4.2. [c] Final pH = 4.2;4.2;4.2. [d] Final pH = 4.8.

Table II-2. (continued)

Element	Method	Spike Level 1[a] Concentration Recovered (ppm)	Spike Level 1[a] Concentration Added (ppm)	Spike Level 1[a] Mean Spike Recovery (%)	Spike Level 2[b] Concentration Recovered (ppm)	Spike Level 2[b] Concentration Added (ppm)	Spike Level 2[b] Mean Spike Recovery (%)	Spike Level 3[c] Concentration Recovered (ppm)	Spike Level 3[c] Concentration Added (ppm)	Spike Level 3[c] Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Cr	AAS	1.28;1.21;1.31 (1.27±0.05)	3.60	35±1	1.00;1.03;1.04 (1.02±0.02)	2.50	41±1	0.29;0.36;0.39 (0.35±0.05)	1.25	28±4	<0.10
	ICP	1.14;1.05;1.13 (1.11±0.05)	3.60	31±2	0.83;0.89;0.90 (0.87±0.04)	2.50	35±2	0.26;0.29;0.32 (0.29±0.03)	1.25	23±3	0.035
	Comb.	1.19±0.10	3.60	33±3	0.95±0.09	2.50	38±4	0.32±0.05	1.25	26±4	
Cu	AAS	2.56;2.51;2.58 (2.55±0.04)	2.70	95±2	1.79;1.77;1.79 (1.78±0.01)	1.80	99±1	0.77;0.81;0.81 (0.79±0.02)	0.90	89±2	<0.050
	ICP	2.62;2.50;2.56 (2.56±0.06)	2.70	95±2	1.76;1.78;1.83 (1.79±0.04)	1.80	100±2	0.77;0.81;0.81 (0.80±0.02)	0.90	89±2	0.049
	Comb.	2.56±0.04	2.70	95±2	1.79±0.02	1.80	99±1	0.80±0.02	0.90	89±2	
Fe	AAS	2.22;18.9;22.2 21.1±1.9)	36.5	58±5	3.97;4.83;4.95 (4.58±0.53)	24	19±2	<0.10;<0.10; <0.10 (<0.10)	12	<1	0.50
	ICP	21.5;17.6;21.0 (20.0±2.1)	36.5	55±6	4.12;4.62;4.62 (4.45±0.29)	24	18±1	<0.10;<0.10; <0.10 (<0.10)	12	<1	0.76
	Comb.	20.6±1.9	36.5	57±5	4.52±0.39	24	19±2	<0.10	12	<1	
Pb	AAS	<0.10;<0.10; <0.10 (<0.10)	0.50	<20	<0.10;<0.10; <0.10 (<0.10)	0.35	<29	<0.10;<0.10; <0.10 (<0.10)	0.175	<57	<0.10
	ICP	<0.20;<0.20; <0.20 (<0.20)	0.50	<40	<0.20;<0.20; <0.20 (<0.20)	0.35	<57	<0.20;<0.20; <0.20 (<0.20)	0.175	NC	<0.20
	Comb.	<0.10	0.50	<20	<0.10	0.35	<29	<0.10	0.175	<57	
Mn	AAS	1.10;1.12;1.12 (1.11±0.01)	1.00	111±1	1.56;1.54;1.55 (1.55±0.01)	1.40	111±1	0.77;0.78;0.79 (0.78±0.01)	0.75	104±1	0.057
	ICP	1.01;0.98;0.99 (0.99±0.02)	1.00	99±2	1.41;1.43;1.44 (1.43±0.02)	1.40	102±1	0.74;0.76;0.76 (0.75±0.01)	0.75	100±1	0.076
	Comb.	1.05±0.07	1.00	105±7	1.49±0.07	1.40	106±5	0.77±0.02	0.75	102±2	
Hg	AAS	0.0028;0.0023; 0.0025 (0.0025±0.0003)	0.0020	127±13	0.0011;0.0009; 0.0009 (0.0010±0.0001)	0.0010	97±12	0.0003;0.0002 0.0002 (0.0002±0.0001)	0.0005	47±12	<0.0001
	ICP	--	0.0020	--	--	0.0010	--	--	0.0005	--	--
	Comb.	0.0025±0.0003	0.0020	127±13	0.0010±0.0001	0.0010	97±12	0.0002±0.0001	0.0005	47±12	

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[a] Final pH = 4.3;4.3;4.3. [b] Final pH = 4.2;4.2;4.2. [c] Final pH = 4.2;4.2;4.2. [d] Final pH = 4.8.

NC = Not calculated.

Table H-2. (continued)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Ni	AAS	0.99;0.91;0.93 (0.94±0.04)	1.40	67±3	0.97;0.95;0.93 (0.95±0.02)	0.90	106±3	0.48;0.48;0.46 (0.47±0.01)	0.50	95±2	<0.050
	ICP	1.04;0.97;1.00 (100±0.035)	1.40	71±3	1.00;1.00;1.01 (1.00±0.01)	0.90	111±1	0.57;0.59;0.59 (0.58±0.01)	0.50	117±2	<0.050
	Comb.	0.97±0.05	1.40	69±3	0.98±0.03	0.90	109±4	0.53±0.06	0.50	106±12	
Se	AAS	0.132;0.088; 0.107 (0.109±0.022)	0.250	44±9	0.033;0.040; 0.038 (0.037±0.004)	0.200	19±2	<0.010;<0.010 <0.010 (<0.010)	0.100	<10	0.008
	ICP	<0.200;<0.200; <0.200 (<0.200)	0.250	<80	<0.200;<0.200; <0.200 (<0.200)	0.200	<100	<0.200;<0.200; <0.200 (<0.200)	1.100	NC	<0.200
	Comb.	0.109±0.022	0.250	44±9	0.037±0.004	0.200	19±2	<0.010	0.100	<10	
Ag	AAS	0.53;0.50;0.85 (0.63±0.19)	3.0	21±6	0.047;0.059; 0.082 (0.063±0.018)	2.0	3±1	0.030;0.034; 0.027 (0.030±0.004)	1.0	3±0	<0.010
	ICP	0.17;0.24;0.60 (0.34±0.23)	3.0	11±8	0.072;0.041; 0.043 (0.052±0.017)	2.0	3±1	0.029;0.030; 0.030 (0.030±0.001)	1.0	3±0	<0.010
	Comb.	0.48±0.25	3.0	16±8	0.057±0.017	2.0	3±1	0.030±0.002	1.0	3±0	
Tl	AAS	2.48;2.42;2.38 (2.43±0.05)	3.0	81±2	1.06;1.00;1.03 (1.03±0.03)	1.5	69±2	0.40;0.37;0.40 (0.39±0.02)	0.75	52±2	<0.20
	ICP	2.07;2.13;2.06 (0.209±0.04)	3.0	70±1	0.91;0.90;0.95 (0.92±0.03)	1.5	61±2	0.35;0.17;0.38 (0.30±0.11)	0.75	40±15	<0.25
	Comb.	2.26±0.19	3.0	75±6	0.98±0.07	1.5	65±4	0.35±0.09	0.75	46±12	
Zn	AAS	2.27;2.27;2.31 (2.28±0.02)	2.25	102±1	1.63;1.59;1.59 (1.60±0.02)	1.5	107±2	0.70;0.78;0.72 (0.73±0.04)	0.75	98±6	0.099
	ICP	2.32;2.26;2.31 (2.30±0.03)	2.25	102±2	1.59;1.56;1.59 (1.58±0.02)	1.5	105±1	0.72;0.80;0.73 (0.75±0.04)	0.75	100±6	0.115
	Comb.	2.29±0.03	2.25	102±1	1.59±0.02	1.5	106±2	0.74±0.04	0.75	99±5	

[a] Final pH = 4.3;4.3;4.3. [b] Final pH = 4.2;4.2;4.2. [c] Final pH = 4.2;4.2;4.2. [d] Final pH = 4.8.

NC = Not calculated.

TABLE H-3. SUMMARY OF RECOVERIES OF METALS FROM THE 50% "BLANK SPIKE" FOR FLY ASH (LOW pH)

Element	Concentration Added (ppm)	Recovery (%)
Al	35	97
Sb	2.5	110
As	0.90	107
Ba	4.75	103
Be	0.10	106
Cd	0.50	106
Cr	1.25	110
Cu	0.90	106
Fe	12	104
Pb	0.175	93
Mn	0.75	103
Ni	0.50	110
Se	0.10	90
Ag	1.0	4
Tl	0.75	93
Zn	0.75	120

TABLE H-4. SUMMARY OF RECOVERIES OF METALS FROM THE 50% "BLANK SPIKE" FOR FLY ASH (pH 5)

Element	Concentration Added (ppm)	Recovery (%)
Al	35.0	86
Sb	2.5	100
As	0.900	99
Ba	4.75	104
Be	0.100	92
Cd	0.500	98
Cr	1.25	102
Cu	0.900	99
Fe	12.0	96
Pb	0.175	103
Mn	0.750	104
Hg	0.0005	320
Ni	0.500	88
Se	0.100	102
Ag	1.00	6
Tl	0.750	97
Zn	0.750	104

TABLE H-5. SUMMARY OF RECOVERIES OF METALS FROM THE 100% "BLANK SPIKE" FOR FLY ASH (pH 5)

Element	Concentration Added (ppm)	Recovery (%)
Al	70.0	90
Sb	5.00	100
As	1.75	95
Ba	9.50	102
Be	0.200	94
Cd	1.00	102
Cr	2.50	104
Cu	1.80	99
Fe	24.0	95
Pb	0.350	101
Mn	1.40	104
Hg	0.0010	280
Ni	0.900	90
Se	0.200	99
Ag	2.00	47
Tl	1.50	95
Zn	1.50	100

TABLE H-6. SUMMARY OF RECOVERIES OF METALS FROM THE 150% "BLANK SPIKE" FOR FLY ASH (pH 5)

Element	Concentration Added (ppm)	Recovery (%)
Al	105	91
Sb	10.0	103
As	2.60	106
Ba	14.25	97
Be	0.300	97
Cd	1.50	100
Cr	3.60	103
Cu	2.70	99
Fe	36.5	99
Pb	0.500	102
Mn	1.00	105
Hg	0.0020	255
Ni	1.40	62
Se	0.250	108
Ag	3.00	14
Tl	3.00	100
Zn	2.25	101

APPENDIX I

RAW DATA FROM PHASE 3 ANALYSES OF EPA MUNICIPAL DIGESTED SLUDGE

This appendix summarizes results of Phase 3 analyses of EPA Municipal Digested Sludge. The sample of sludge was dried, ground, and homogenized prior to use for Phase 3 extractions and bulk analysis. Bulk concentrations of the elements of interest were determined by ERCO and verified by an outside laboratory.

Each sample ws prepared for the EP extraction according to methods outlined by EPA[1]. After addition of distilled water to the dried sludge, the samples were spiked in triplicate at three different concentration levels representing 150%, 100%, and 50% of the total metal concentrations determined from the bulk analysis. For each spike level, a "blank spike" was prepared by adding the spikes to distilled water. This solution was utilized to verify the integrity of the sample spikes. Raw data for the Low pH extracts are presented in Table I-1.

Raw data for the pH 5 extracts are presented in Table I-2. For each element presented in Tables I-1 and I-2, individual determinations performed by AAS and ICP are presented, along with the mean and standard deviation calculated from the triplicate determinations. A combined mean and standard deviation from the AAS and ICP data is also presented. Recoveries of metal spikes from the 50%, 100%, and 150% "blank spikes" for the EPA Municipal Digested Sludge are presented in Tables I-3, I-4, and I-5, respectively.

TABLE I-1. SUMMARY OF PHASE 3 ANALYSES OF MUNICIPAL DIGESTED SLUDGE (LOW pH)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Low pH Extract (ppm)[d]
Al	AAS	75; 75; 83 (78 ± 5)	15	520 ± 33	7.8; 9.2; 7.8 (8.3 ± 0.8)	10	83 ± 8	50; 56; 74 (60 ± 12)	5.0	1200 ± 240	3.36
	ICP	67; 63; 62 (64 ± 3)	15	427 ± 20	8.0; 8.0; 8.8 (8.3 ± 0.5)	10	83 ± 5	36; 41; 56 (44 ± 10)	5.0	880 ± 200	3.90
	Comb.	71 ± 8	15	473 ± 53	8.3 ± 0.6	10	83 ± 6	52 ± 13	5.0	1040 ± 260	--
Sb	AAS	0.93; 1.26; 1.2 (1.1 ± 0.56)	7.5	15 ± 7.5	0.93; 0.69; 0.93 (0.85 ± 0.14)	5.0	17 ± 3	<0.5; <0.5; <0.5	2.5	<20	<0.5
	ICP	1.5; 1.0; 1.5 (1.3 ± 0.28)	7.5	17 ± 4	1.25; 1.25; 0.75 (1.08 ± 0.25)	5.0	22 ± 5	<0.15; <0.15; <0.15	2.5	<6	0.15
	Comb.	1.2 ± 0.24	7.5	16 ± 3	0.97 ± 0.24	5.0	19 ± 5		2.5		--
As	AAS	0.205; 0.200; 0.190 (0.198 ± 0.008)	0.235	84 ± 3	0.083; 0.071; 0.081 (0.078 ± 0.006)	0.125	62 ± 5	0.047; 0.141; 0.081 (0.097 ± 0.038)	0.0625	155 ± 61	0.065
	ICP	<0.2; <0.2; <0.2	0.235	NC	<0.2; <0.2; <0.2	0.125	NC	<0.2; <0.2; <0.2	0.0625	NC	0.177
	Comb.	0.198 ± 0.008	0.235	84 ± 3	0.078 ± 0.006	0.125	62 ± 5	0.097 ± 0.038	0.0625	115 ± 61	--
Ba	AAS	0.93; 0.93; 0.93 (0.93 ± 0)	11.5	8 ± 0	1.57; 1.57; 1.76 (1.63 ± 0.10)	7.6	21 ± 1	1.29; 1.29; 1.29 (1.29 ± 0)	3.0	43 ± 0	13
	ICP	0.73; 0.93; 0.73 (0.80 ± 0.12)	11.5	7 ± 1	0.75; 0.75; 0.75 (0.75 ± 0)	7.6	10 ± 0	1.0; 1.0; 1.0 (1.00 ± 0)	3.0	26 ± 0	0.77
	Comb.	0.86 ± 0.10	11.5	7.5 ± 1	1.20 ± 0.49	7.6	16 ± 6	1.16 ± 0.16	3.0	30 ± 4	--
Be	AAS	<0.025;<0.025;<0.025	0.0075	NC	<0.025;<0.025;<0.025	0.005	NC	<0.025;<0.025;<0.025	0.0025	NC	<0.025
	ICP	<0.125;<0.125;<0.125	0.0075	NC	<0.125;<0.125;<0.125	0.005	NC	<0.125;<0.125;<0.125	0.0025	NC	<0.125
	Comb.	<0.025	0.0075	NC		0.005	NC		0.0025	NC	--
Cd	AAS	1.05; 0.95; 0.95 (0.98 ± 0.06)	0.55	178 ± 11	0.75; 0.75; 0.75 (0.75 ± 0)	0.375	200 ± 0	0.45; 0.45; 0.45 (0.45 ± 0)	0.20	225 ± 0	0.55
	ICP	1.28; 1.28; 1.28 (1.28 ± 0)	0.55	233 ± 0	0.80; 0.80; 0.80 (0.80 ± 0)	0.375	213 ± 0	0.43; 0.43; 0.43 (0.43 ± 0)	0.20	215 ± 0	0.57
	Comb.	1.13 ± 0.17	0.55	205 ± 31	0.78 ± 0.03	0.375	208 ± 8	0.44 ± 0.01	0.20	220 ± 5	--
Cr	AAS	12.7; 12.7; 11.7 (12.4 ± 0.6)	6.25	198 ± 10	5.87; 6.17; 5.87 (5.97 ± 0.17)	4.0	149 ± 4	4.47; 4.47; 4.47 (4.47 ± 0)	2.0	224 ± 0	0.33
	ICP	12.8; 12.3; 12.0 (12.4 ± 0.4)	6.25	198 ± 6	6.38; 6.28; 6.28 (6.31 ± 0.06)	4.0	158 ± 1.5	3.78; 4.08; 4.28 (4.05 ± 0.25)	2.0	203 ± 13	0.22
	Comb.	12.4 ± 0.45	6.25	198 ± 7	6.14 ± 0.22	4.0	154 ± 6	4.26 ± 0.28	2.0	213 ± 14	--
Cu	AAS	68.5; 66.5; 65.5 (66.8 ± 1.5)	35	191 ± 4	7.5; 10.5; 7.5 (8.5 ± 1.7)	2.5	340 ± 68	30.5; 32.5; 36.5 (33 ± 3)	12.5	264 ± 24	9.5
	ICP	77; 77; 73 (76 ± 2)	35	217 ± 6	8.5; 8.5; 8.5 (8.5 ± 0)	2.5	340 ± 0	32; 35; 40 (36 ± 4)	12.5	288 ± 32	10.0
	Comb.	71 ± 5	35	203 ± 14	8.5 ± 1.1	2.5	340 ± 44	34 ± 3.5	12.5	272 ± 28	--

[a] Final pH = 2.26; 2.27; 2.27. [b] Final pH = 3.21; 3.20; 3.26. [c] Final pH = 2.41; 2.40; 2.22. [d] Final pH = 3.03.

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TABLE I-1. Continued.

Element	Method	Spike Level 1[a] Concentration Recovered (ppm)	Spike Level 1[a] Concentration Added (ppm)	Spike Level 1[a] Mean Spike Recovery (%)	Spike Level 2[b] Concentration Recovered (ppm)	Spike Level 2[b] Concentration Added (ppm)	Spike Level 2[b] Mean Spike Recovery (%)	Spike Level 3[c] Concentration Recovered (ppm)	Spike Level 3[c] Concentration Added (ppm)	Spike Level 3[c] Mean Spike Recovery (%)	Concentration in Low pH Extract (ppm)[d]
Fe	AAS	207; 177; 167 (184 ± 21)	60	307 ± 35	77; 82; 81 (80 ± 3)	40	200 ± 8	25; 0.3; 20 (15 ± 3)	20	75 ± 65	3.0
	ICP	272; 240; 240 (251 ± 18)	60	418 ± 30	90; 90; 90 (90 ± 0)	40	225 ± 0	29; 7; 24 (20 ± 12)	20	100 ± 60	3.0
	Comb.	217 ± 41	60	362 ± 68	85 ± 6	40	213 ± 15	17.6 ± 11	20	88 ± 55	--
Pb	AAS	0.51; 0.39; 0.51 (0.47 ± 0.07)	16	2.9 ± 0.4	<0.25; <0.25; <0.25	10.5	<2.4	1.0; 1.1; 2.1 (1.4 ± 0.6)	5.25	27 ± 11	<0.25
	ICP	1.0; <0.2; <0.2	16	NC	<0.2; <0.2; <0.2	10.5	<2	0.5; <0.2; 1.25	5.25	NC	<0.2
	Comb.	0.47 ± 0.07	16	2.9 ± 0.4		10.5	<2	1.4 ± 0.6	5.25	27 ± 11	--
Mn	AAS	13.6; 12.6; 13.6 (13.3 ± 0.6)	6.5	205 ± 9	0.6; 0.6; 0.6 (0.6 ± 0)	4.5	191 ± 0	5.6; 5.6; 5.6 (5.6 ± 0)	2.25	249 ± 0	6.4
	ICP	13.5; 13.5; 12.5 (13.2 ± 0.6)	6.5	203 ± 9	0.5; 0.5; 0.5 (0.5 ± 0)	4.5	189 ± 0	4.5; 5.5; 5.5 (5.1 ± 0.6)	2.25	249 ± 27	6.5
	Comb.	13.2 ± 0.5	6.5	203 ± 8	0.6 ± 0.06	4.5	191 ± 1	5.4 ± 0.4	2.25	240 ± 18	--
Hg	AAS	.00093;.00075;.00075 (0.00081 ± 0.00010)	0.0020	41 ± 5	0.0012;0.0010;0.0011 (0.0011 ± 0.00010)	0.0010	110 ± 10	.00092;.00084;.00066 (0.00081 ± 0.00013)	0.0005	162 ± 26	0.00024
	ICP	<10	0.0020	NC	<10	0.0010	NC	<10	0.0005	NC	<10
	Comb.	0.00081 ± 0.00010	0.0020	41 ± 5	0.0011 ± 0.00010	0.0010	110 ± 10	0.00081 ± 0.00013	0.0005	162 ± 26	--
Ni	AAS	18.5; 18.5; 15.5 (17.5 ± 1.7)	5.5	318 ± 31	17.5; 14.5; 16.5 (16.2 ± 1.5)	3.75	432 ± 40	7.5; 13.5; 13.5 (11.5 ± 3.5)	1.07	615 ± 187	4.5
	ICP	12.8; 12.3; 11.8 (12.3 ± 0.5)	5.5	224 ± 9	7.8; 7.5; 7.5 (7.6 ± 0.2)	3.75	203 ± 5	5.0; 5.0; 4.8 (4.9 ± 0.1)	1.07	262 ± 5	5.74
	Comb.	14.9 ± 3	5.5	271 ± 3	11.9 ± 48	3.75	317 ± 128	5.3 ± 4.2	1.07	283 ± 225	--
Se	AAS	<0.01; <0.01; <0.01	0.125	<8	<0.01; <0.01; <0.01	0.075	<13	<0.01; <0.01; <0.01	0.0375	<27	0.013
	ICP	<0.1; <0.1; <0.1	0.125	NC	<0.1; <0.1; <0.1	0.075	NC	<0.1; <0.1; <0.1	0.0375	NC	<0.1
	Comb.	<0.01	0.125	<8	<0.01	0.075	<13	<0.01	0.0375	<27	--
Ag	AAS	0.028; 0.026; 0.028 (0.027 ± 0.001)	1.6	1.7 ± 0.1	<0.015;<0.015;<0.015	1.25	<1.2	<0.015;<0.015;<0.015	0.625	<2.4	<0.015
	ICP	<0.05; <0.05; <0.05	1.6	<3	<0.05; <0.05; <0.05	1.25	<4	<0.05; <0.05; <0.05	0.625	<8	0.006
	Comb.	0.027 ± 0.001	1.6	1.7 ± 0.1	<0.015	1.25	<1.2	<0.015	0.625	<2.4	--
Tl	AAS	1.1; 1.1; 1.1 (1.1 ± 0)	1.0	110 ± 0	0.38; 0.38; 0.38 (0.38 ± 0)	0.75	51 ± 0	0.53; 0.27; <0.25 (0.35 ± 0.16)	0.375	93 ± 43	<0.25
	ICP	3.5; 2.0; 1.7 (2.4 ± 1.0)	1.0	240 ± 100	2.2; 2.2; 2.0 (2.1 ± 0.1)	0.75	280 ± 13	0.97; 1.47; 1.72 (1.39 ± 0.38)	0.375	371 ± 101	0.20
	Comb.	1.75 ± 0.94	1.0	175 ± 94	1.26 ± 0.96	0.75	168 ± 128	0.87 ± 0.62	0.375	232 ± 165	--
Sn	AAS	86; 76; 76 (79 ± 6)	44.0	180 ± 14	46; 38; 38 41 ± 4.6)	26.0	158 ± 18	29; 42; 40 (37 ± 7)	13.0	285 ± 54	44
	ICP	99.5; 96.5; 94.5 (96.8 ± 2.5)	44.0	220 ± 6	56; 56; 50 (54 ± 4)	26.0	208 ± 15	35; 40; 42 (39 ± 4)	13.0	300 ± 31	40.5
	Comb.	88 ± 10	44.0	200 ± 23	47 ± 8	26.0	181 ± 31	38 ± 5	13.0	292 ± 38	--

[a] Final pH = 2.26; 2.27; 2.27. [b] Final pH = 3.21; 3.20; 3.26. [c] Final pH = 2.41; 2.40; 2.22. [d] Final pH = 3.03.

TABLE I-2. SUMMARY OF PHASE 3 ANALYSES OF MUNICIPAL DIGESTED SLUDGE (pH 5)

		Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			
Element	Method	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Al	AAS	<0.50;<0.50; <0.50 (<0.50)	15	<3	<0.50;<0.50; <0.50 (<0.50)	10	<5	<0.50;<0.50; <0.50 (<0.50)	5	<10	<0.50
	ICP	0.101;0.096; 0.076 (0.091±0.013)	15	1±0	0.073;0.083; 0.063 (0.073±0.010)	10	1±0	0.113;0.162 0.087 (0.121±0.038)	5	2±1	0.078
	Comb.	0.091±0.013	15	1±0	0.073±0.010	10	1±0	0.121±0.038	5	2±1	
Sb	AAS	0.58;0.64;0.58 (0.060±0.03)	7.5	8±1	0.26;0.35;0.41 (0.34±0.08)	5.0	7±2	<0.20;<0.20; <0.20 (<0.20)	2.5	<8	<0.20
	ICP	0.60;0.62;0.64 (0.62±0.02)	7.5	8±1	0.30;0.35;0.44 (0.36±0.07)	5.0	7±2	0.19;0.19;0.16 (0.18±0.02)	2.5	7±1	<0.20
	Comb.	0.61±0.03	7.5	8±1	0.35±0.07	5.0	7±2	0.18±0.02	2.5	7±1	
As	AAS	<0.100;<0.100; <0.100 (<0.100)	0.24	<42	<0.100;<0.100; <0.100 (<0.100)	0.125	<80	<0.100;<0.100; <0.100 (<0.100)	0.060	NC	<0.010
	ICP	<0.200;<0.200; <0.200 (<0.200)	0.24	<83	<0.200;<0.200; <0.200 (<0.200)	0.125	NC	<0.200;<0.200; <0.200 (<0.200)	0.060	NC	<0.200
	Comb.	<0.100	0.24	<42	<0.100	0.125	<80	<0.100	0.060	NC	
Ba	AAS	1.3;1.2;1.3 (1.3±0.1)	11.5	11±1	0.66;0.51;0.51 (0.56±0.09)	7.6	8±1	<0.50;<0.50; <0.50 (<0.50)	3.8	<13	<0.50
	ICP	0.71;0.77;0.79 (0.76±0.04)	11.5	7±1	0.40;0.36;0.38 (0.37±0.01)	7.6	5±0	0.14;0.15;0.20 (0.16±0.03)	3.8	4±1	0.038
	Comb.	1.01±0.28	11.5	9±2	0.47±0.11	7.6	7±2	0.16±0.03	3.8	4±1	
Be	AAS	<0.010;<0.010; <0.010 (<0.010)	0.375	<3	<0.010;<0.010; <0.010 (<0.010)	0.250	<4	<0.010;<0.010 <0.010 (<0.010)	0.125	<8	<0.010
	ICP	<0.010;<0.010; <0.010 (<0.010)	0.375	<3	<0.010;<0.010; <0.010 (<0.010)	0.250	<4	<0.010;<0.010 <0.010 (<0.010)	0.125	<8	<0.010
	Comb.	<0.010	0.375	<3	<0.010	0.250	<4	<0.010	0.125	<8	
Cd	AAS	0.18;0.18;0.18 (0.18±0.00)	0.55	33±0	0.10;0.10;0.10 (0.10±0.00)	0.375	27±0	0.063;0.063; 0.053 (0.060±0.006)	0.20	30±3	0.057
	ICP	0.14;0.15;0.15 (0.15±0.01)	0.55	26±1	0.076;0.073; 0.081 (0.077±0.004)	0.375	20±2	0.048;0.045 0.037 (0.043±0.006)	0.20	22±3	0.063
	Comb.	0.16±0.02	0.55	30±4	0.088±0.013	0.375	24±4	0.052±0.010	0.20	26±5	

---

[a] Final pH = 5.1;5.1;5.1. [b] Final pH = 5.1;5.1;5.1. [c] Final pH = 5.1;5.2;5.2. [d] Final pH = 5.1.

NC = Not calculated.

Table I-2. (continued)

Element	Method	Spike Level 1[a]			Spike Level 2[b]			Spike Level 3[c]			Concentration in Unspiked EP Extract (ppm)[d]
		Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	Concentration Recovered (ppm)	Concentration Added (ppm)	Mean Spike Recovery (%)	
Cr	AAS	0.39;0.36;0.36 (0.37±0.02)	6.25	6±0	0.24;0.22;0.21 (0.22±0.02)	4.0	6±1	0.13;0.14;0.13 (0.14±0.01)	2.0	7±0	<0.10
	ICP	0.29;0.26;0.28 (0.28±0.02)	6.25	4±1	0.19;0.17;0.17 (0.18±0.01)	4.0	4±1	0.11;0.12;0.11 (0.11±0.01)	2.0	6±0	<0.025
	Comb.	0.32±0.05	6.25	5±1	0.20±0.03	4.0	5±1	0.12±0.01	2.0	6±1	
Cu	AAS	2.94;2.68;2.90 (2.84±0.14)	35.0	8±0	1.60;1.47;1.58 (1.55±0.07)	25.0	6±0	0.83;0.75;0.64 (0.74±0.10)	12.5	6±1	0.87
	ICP	2.81;2.48;2.70 (2.66±0.17)	35.0	8±1	1.58;1.38;1.51 (1.49±0.10)	25.0	6±0	0.78;0.75;0.61 (0.71±0.09)	12.5	6±1	0.89
	Comb.	2.75±0.17	35.0	8±1	1.52±0.08	25.0	6±0	0.73±0.08	12.5	6±1	
Fe	AAS	<0.10;<0.10; <0.10 (<0.10)	60	<1	<0.10;<0.10; <0.10 (<0.10)	40	<1	0.19;0.54;0.23 (0.32+0.19)	20	2±1	0.58
	ICP	<0.10;<0.10; <0.10 (<0.10)	60	<1	<0.10;<0.10; <0.10 (<0.10)	40	<1	0.18;0.53;0.24 (0.32+0.19)	20	2+1	0.52
	Comb.	<0.10	60	<1	<0.10	40	<1	0.32±0.17	20	2±1	
Pb	AAS	<0.10;<0.10; <0.10 (<0.10)	16	<1	<0.10;<0.10; <0.10 (<0.10)	10.5	<1	<0.10;<0.10; <0.10 (<0.10)	5.25	<2	<0.10
	ICP	<0.20;<0.20; <0.20 (<0.20)	16	<1	<0.20;<0.20; <0.20 (<0.20)	10.5	<2	<0.20;<0.20 <0.20 (<0.20)	5.25	<4	<0.20
	Comb.	<0.10	16	<1	<0.10	10.5	<1	<0.10	5.25	<2	
Mn	AAS	4.43;4.55;4.43 (4.47±0.07)	6.5	69±1	2.69;2.69;2.81 (2.73±0.07)	4.5	61±1	1.45;1.45;1.45 (1.45±0.00)	2.25	64±0	1.36
	ICP	3.74;3.75;3.76 (3.75±0.01)	6.5	58±0	2.44;2.41;2.50 (2.45±0.05)	4.5	55±1	1.22;1.26;1.25 (1.24±0.2)	2.25	55±1	1.29
	Comb.	4.11±0.40	6.5	63±6	2.59±0.16	4.5	58±3	1.35+0.11	2.25	60+5	
Hg	AAS	0.0037;0.0041; 0.0032 (0.0037±0.0005)	0.0020	183±23	0.0041;0.0041 0.0035 (0.0039±0.0003)	0.0010	390±35	0.0054;0.0044; 0.0039 (0.0046±0.0008)	0.0005	913±153	0.0044
	ICP	--	0.0020	--	--	0.0010	--	--	0.0005	--	--
	Comb.	0.0037±0.0005	0.0020	183±23	0.0039±0.0003	0.0010	390±35	0.0046±0.0008	0.0005	913±153	

[a] Final pH = 5.1;5.1;5.1. [b] Final pH = 5.1;5.1;5.1. [c] Final pH = 5.1;5.2;5.2. [d] Final pH = 5.1.

NC = Not calculated.

Table I-2. (continued)

Element	Method	Spike Level 1[a] Concentration Recovered (ppm)	Spike Level 1[a] Concentration Added (ppm)	Spike Level 1[a] Mean Spike Recovery (%)	Spike Level 2[b] Concentration Recovered (ppm)	Spike Level 2[b] Concentration Added (ppm)	Spike Level 2[b] Mean Spike Recovery (%)	Spike Level 3[c] Concentration Recovered (ppm)	Spike Level 3[c] Concentration Added (ppm)	Spike Level 3[c] Mean Spike Recovery (%)	Concentration in Unspiked EP Extract (ppm)[d]
Ni	AAS	3.11;3.11;3.31 (3.18±0.12)	5.50	58+2	2.09;1.68;1.88 (1.88±0.21)	3.75	50+6	1.00;1.04;0.98 (1.01±0.03)	1.875	53±2	2.53
	ICP	2.64;2.62;2.58 (2.61±0.03)	5.50	48±1	1.71;1.52;1.61 (1.61±0.10)	3.75	43±3	0.82;0.78;0.82 (0.81±0.02)	1.875	43±1	2.48
	Comb.	2.90±0.32	5.50	53±6	1.75±0.21	3.75	47±5	0.91±0.11	1.875	48±6	
Se	AAS	<0.010;<0.010; <0.010 (<0.010)	0.125	<8	<0.010;<0.010; <0.010 (<0.010)	0.075	<13	<0.010;<0.010; <0.010 (<0.010)	0.040	<25	<0.010
	ICP	<0.200;<0.200; <0.200 (<0.200)	0.125	NC	<0.200;<0.200; <0.200 (<0.200)	0.075	NC	<0.200;<0.200; <0.200 (<0.200)	0.040	NC	<0.200
	Comb.	<0.010	0.125	<8	<0.010	0.075	<13	<0.010	0.040	<25	
Ag	AAS	<0.010;<0.010 <0.010 (<0.010)	1.60	<2	<0.010;<0.010 <0.010 (<0.010)	1.25	<1	0.018;0.018; 0.016 (0.017±0.001)	0.625	1±0	0.082
	ICP	<0.010;<0.010 <0.010 (<0.010)	1.60	<2	<0.010;<0.010 <0.010 (<0.010)	1.25	<1	0.024;0.023; 0.015 (0.021±0.005)	0.625	2±1	0.073
	Comb.	<0.010	1.60	<2	<0.010	1.25	<1	0.019±0.004	0.625	2±1	
Tl	AAS	<0.200;<0.20; <0.20 (<0.20)	1.00	<20	<0.20;<0.20; <0.20 (<0.20)	0.750	<27	<0.20;<0.20; <0.20 (<0.20)	0.375	<53	<0.20
	ICP	0.20;0.29;0.28 (0.26±0.05)	1.00	26±5	<0.25;<0.24; <0.25 (<0.25)	0.750	<27	<0.25;<0.25; <0.25 (<0.25)	0.375	<53	<0.25
	Comb.	0.26±0.05	1.00	26±5	<0.20	0.750	<27	<0.20	0.375	<53	
Zn	AAS	15.4; 15.6; 15.4 (15.5±0.1) (15.5±0.1)	44	35±0	7.72; 8.22; 8.62 (8.19±0.45)	26	32±2	4.42; 4.42; 4.42 (4.42±0.00)	13	34±0	5.68
	ICP	13.6; 13.3; 13.1 (13.3±0.3)	44	30±1	6.88; 6.58; 7.28 (6.91+0.35)	26	26±2	3.67; 3.77; 3.81 (3.75+0.07)	13	29±1	5.52
	Comb.	14.4±1.2	44	33±3	7.55±0.78	26	29±3	4.09±0.37	13	31±3	

[a] Final pH = 5.0;5.1;5.1. [b] Final pH = 5.0;5.1;5.1. [c] Final pH = 5.0;5.1;5.1. [d] Final pH = 5.2.

NC = Not calculated.

TABLE I-3. SUMMARY OF RECOVERIES OF METALS FROM THE 50% "BLANK SPIKE" FOR EPA MUNICIPAL DIGESTED SLUDGE

Element	Concentration Added (ppm)	Recovery (%)
Al	5.00	97
Sb	2.50	100
As	0.060	103
Ba	3.80	96
Be	0.125	104
Cd	0.200	100
Cr	2.00	103
Cu	12.5	104
Fe	20.0	88
Pb	5.25	101
Mn	2.25	103
Hg	0.0005	200
Ni	1.875	94
Se	0.040	103
Ag	0.625	6
Tl	0.375	107
Zn	13.0	98

TABLE I-4. SUMMARY OF RECOVERIES OF METALS FROM THE 100% "BLANK SPIKE" FOR EPA MUNICIPAL DIGESTED SLUDGE

Element	Concentration Added (ppm)	Recovery (%)
Al	10.0	99
Sb	5.00	100
As	0.125	106
Ba	7.6	93
Be	0.250	102
Cd	0.375	93
Cr	4.00	102
Cu	25.0	102
Fe	40.0	86
Pb	10.5	94
Mn	4.50	104
Hg	0.0010	200
Ni	3.75	96
Se	0.075	103
Ag	1.25	16
Tl	0.750	103
Zn	26.0	95

TABLE I-5. SUMMARY OF RECOVERIES OF METALS FROM THE 150% "BLANK SPIKE" FOR EPA MUNICIPAL DIGESTED SLUDGE

Element	Concentration Added (ppm)	Recovery (%)
Al	15.0	99
Sb	7.50	101
As	0.235	99
Ba	11.5	90
Be	0.375	95
Cd	0.550	95
Cr	6.25	103
Cu	35.0	100
Fe	60.0	87
Pb	16.0	100
Mn	6.50	102
Hg	0.0020	170
Ni	5.50	95
Se	0.125	105
Ag	1.60	23
Tl	1.00	100
Zn	44.0	99

METHOD 1310

EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD AND STRUCTURAL INTEGRITY TEST

1.0 Scope and Application

1.1 The extraction procedure (EP) described in this method is designed to simulate the leaching a waste will undergo if disposed of in an improperly designed sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphasic samples.

2.0 Summary of Method

2.1 If a representative sample of the waste contains more than 0.5% solids, the solid phase of the sample is extracted with deionized water which is maintained at a pH of 5 ± 0.2 using acetic acid. The extract is analyzed to determine if any of the threshold limits listed in Table 1 are exceeded. Table 1 also specifies the approved method of analysis. Wastes that contain less than 0.5% solids are not subjected to extraction, but are directly analyzed and evaluated in a manner identical to that of extracts.

3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods referenced in Table 1.

4.0 Apparatus and Materials

4.1 Extractor: For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures 1-3 of this method and Section 2.2 (Mobility) of this manual and are available from Associated Designs & Manufacturing Co., Alexandria, Virginia; Kraft Apparatus Inc., Mineola, New York; Millipore, Bedford, Massachusetts; and Rexnard, Milwaukee, Wisconsin.

4.2 pH Meter or pH Controller (Chemtrix, Inc., Hillsboro, Oregon is a possible source of a pH controller).

4.3 Filter holder: A filter holder capable of supporting a 0.45-μ filter membrane and able to withstand the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 5.3 kg/cm^3 (75 psi) of pressure. The type of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table 2.

TABLE 1. MAXIMUM CONCENTRATION OF CONTAMINANTS FOR CHARACTERISTIC OF EP TOXICITY

Contaminant	Maximum concentration (mg/l)	Analytical method
Arsenic	5.0	7060, 7061
Barium	100.0	7080, 7081
Cadmium	1.0	7130, 7131
Total Chromium	5.0	7190, 7191
Hexavalent Chromium	5.0	7195, 7196, 7197
Lead	5.0	7420, 7421
Mercury	0.2	7470
Selenium	1.0	7740, 7741
Silver	5.0	7760, 7761
Endrin (1,2,3,4,10,10-Hexachloro-1 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1 4-endo, endo-5,8-dimethanonaphthalene)	0.02	8080
Lindane (1,2,3,4,5,6-Hexachlorocyclohexane, gamma isomer)	0.4	8080
Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0	8080
Toxaphene ($C_{10}H_{10}Cl_8$, Technical chlorinated camphene, 67-69% chlorine)	0.5	8080
2,4-D (2,4-Dichlorophenoxyacetic acid)	10.0	8150
2,4,5-TP (Silvex) (2,4,5-Trichlorophenoxypropionic acid)	1.0	8150

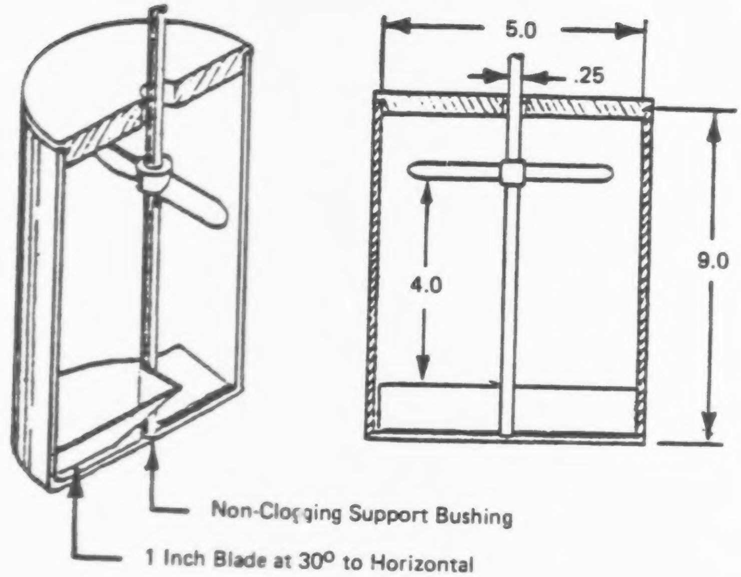

5.0
.25
9.0
4.0
Non-Clogging Support Bushing
1 Inch Blade at 30° to Horizontal

Figure 1. Extractor.

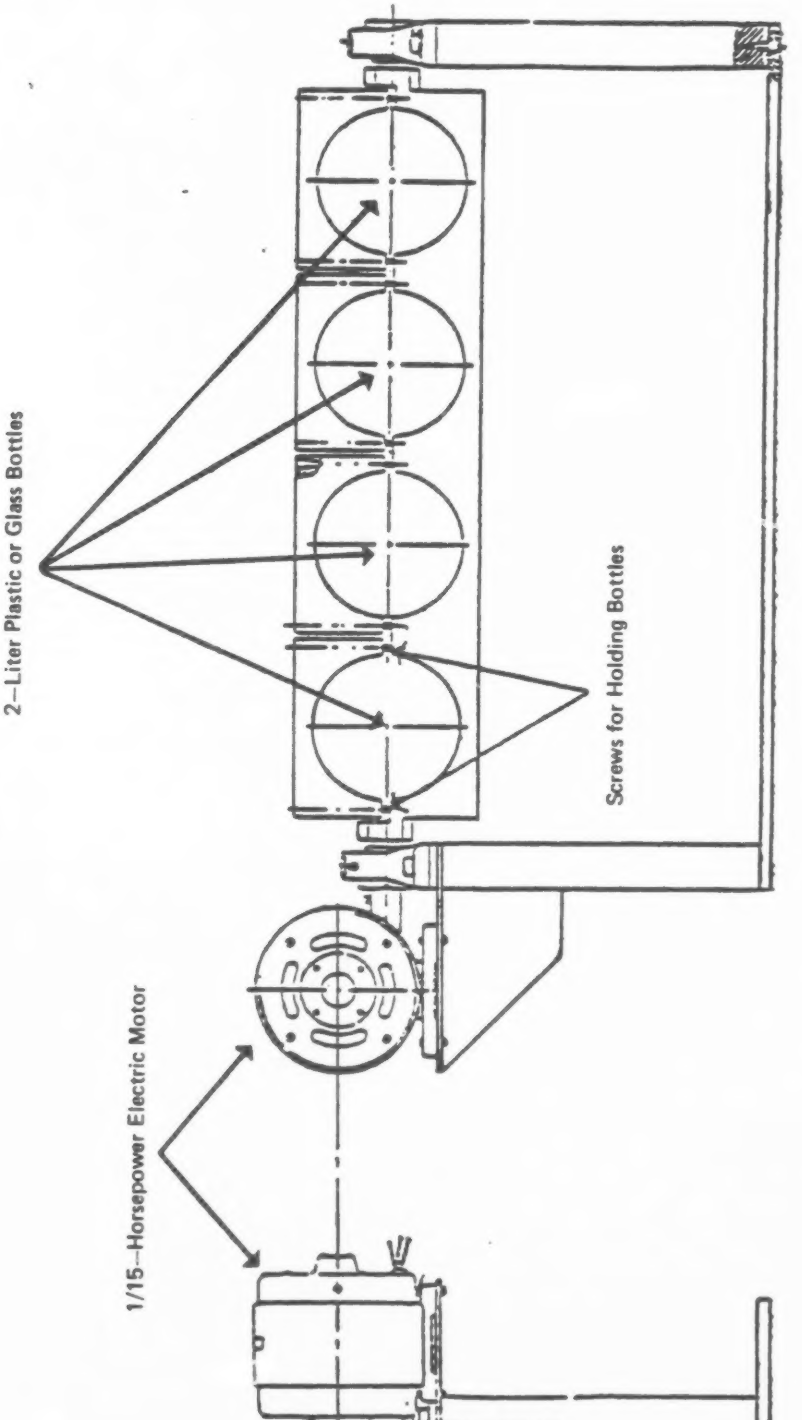

2-Liter Plastic or Glass Bottles
Screws for Holding Bottles
1/15-Horsepower Electric Motor
29 RPM

Figure 2. Rotary Extractor.

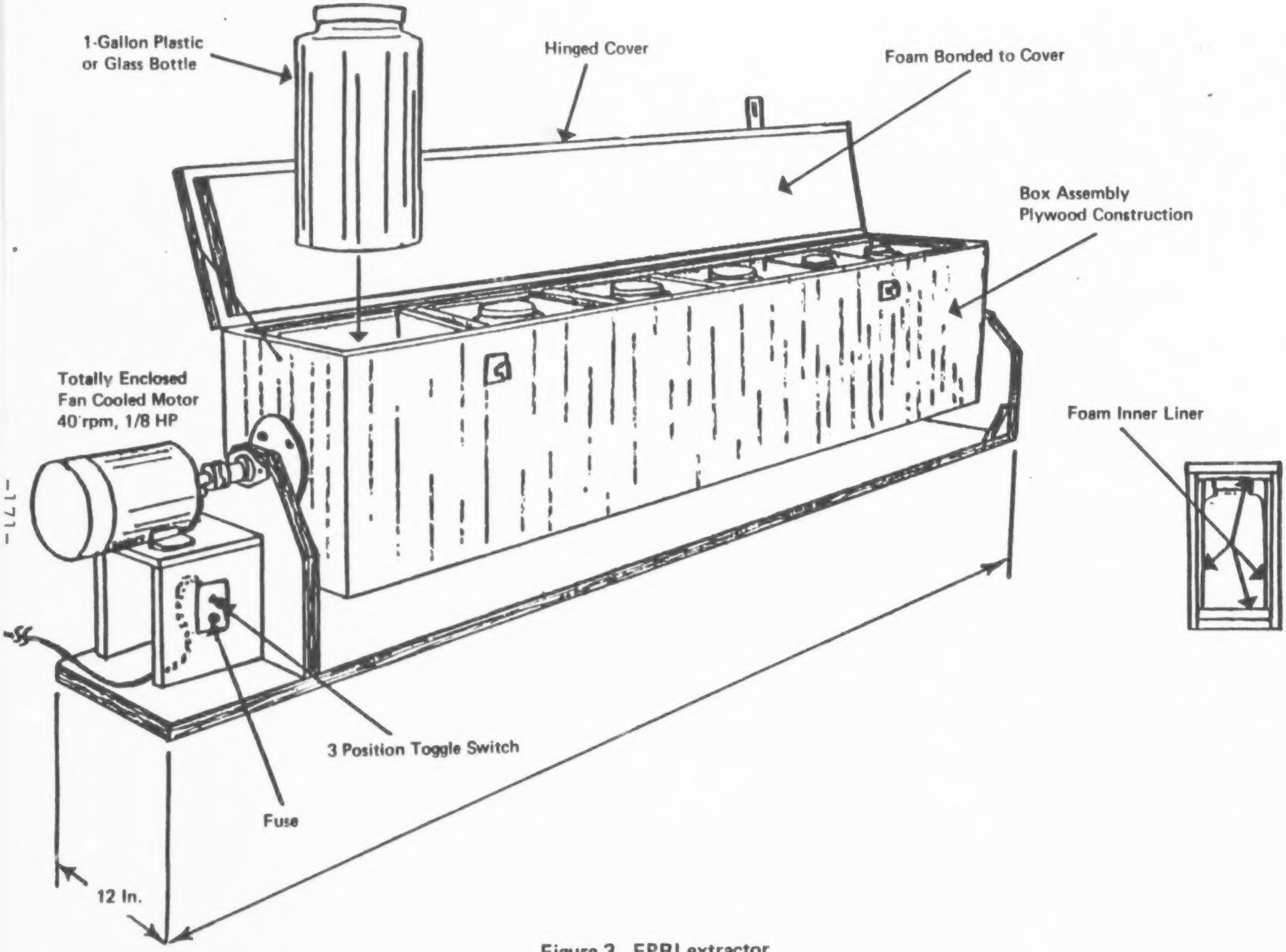

1-Gallon Plastic
or Glass Bottle
Hinged Cover
Foam Bonded to Cover
Box Assembly
Plywood Construction
Totally Enclosed
Fan Cooled Motor
40 rpm, 1/8 HP
Foam Inner Liner
3 Position Toggle Switch
Fuse
12 In.

Figure 3. EPRI extractor.

4.4 Filter membrane: Filter membrane suitable for conducting the required filtration shall be fabricated from a material which: (1) is not physically changed by the waste material to be filtered, and (2) does not absorb or leach the chemical species for which a waste's EP Extract will be analyzed. Table 3 lists filter media known to the agency and generally found to be suitable for solid waste testing.

4.4.1 In cases of doubt, contact the filter manufacturer to determine if the membrane or the prefilter are adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. After 48 hr, a filter that undergoes visible physical change (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

TABLE 2. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
Vacuum Filters			
Nalgene	500 ml	44-0045	Disposable plastic unit, includes prefilter and filter pads, and reservoir; should be used when solution is to be analyzed for inorganic constituents
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
Pressure Filters			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

TABLE 3. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
Coarse Prefilter		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
Medium prefilters		
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
Fine prefilters		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
Fine filters (0.45 μm)		
Gelman	60173, 60177	60540 or 66149, 60544 or 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218[a]
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

[a] Susceptible to decomposition by certain polar organic solvents.

4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and re-analyze. If the concentration of the filtrate differs from the original standard, the filter membrane leaches or absorbs one or more of the chemical species.

4.5 Structural integrity tester: Having a 3.18-cm (1.25-in.) diameter hammer weighing 0.33 kg (0.73 lb) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 4.

5.0 Reagents

5.1 Deionized water: Water should be monitored for impurities.

5.2 0.5 N acetic acid: This can be made by diluting concentrated glacial acetic acid (17.5 N). The glacial acetic acid should be of high purity and monitored for impurities.

5.3 Analytical standards should be prepared according to the analytical methods referenced in Table 1.

6.0 Sample Collection, Preservation and Handling

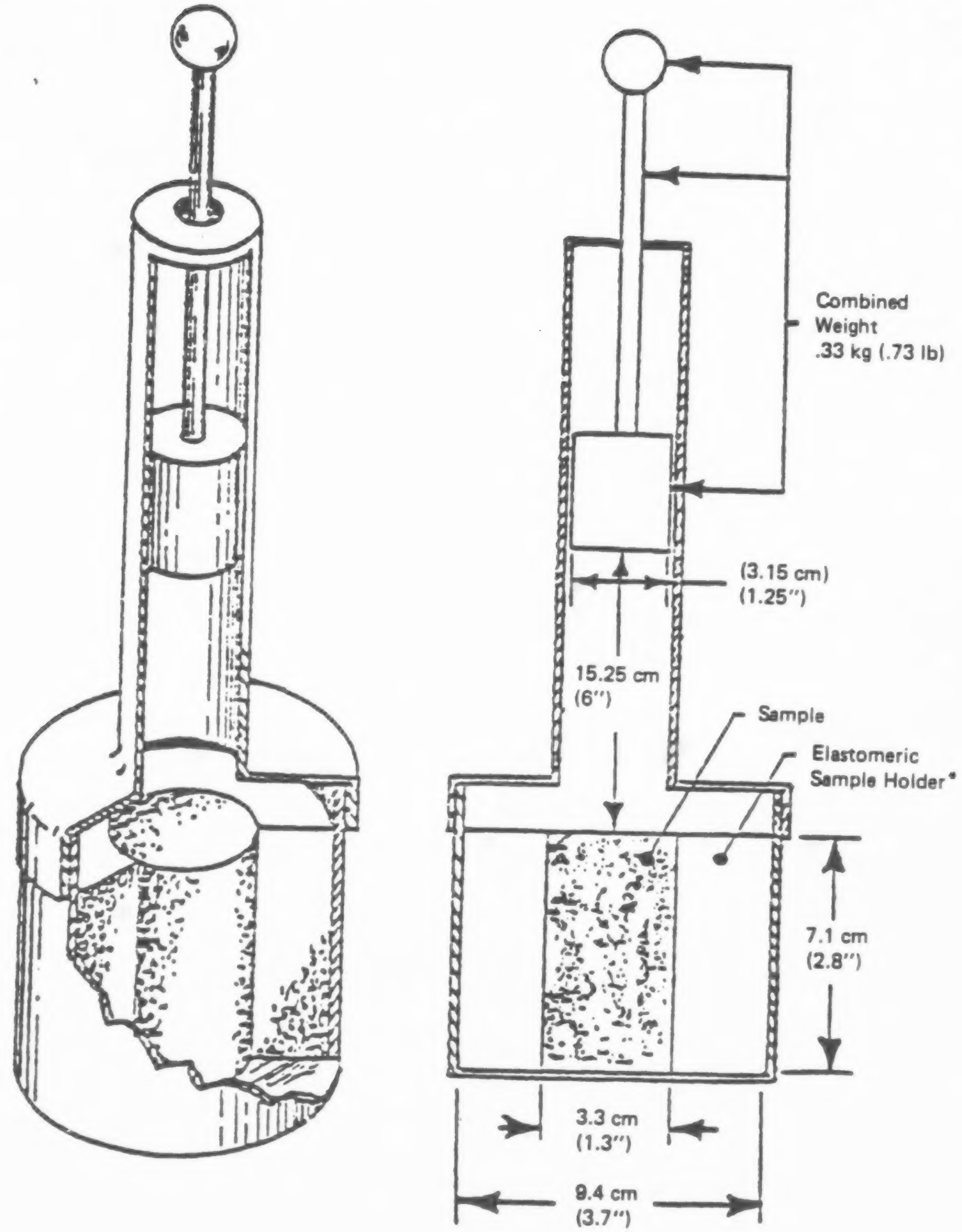
6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 Preservatives must not be added to samples.

6.3 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

7.0 Procedure

7.1 If the waste does not contain any free liquid, go to Section 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to ±0.01 g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipette.


Combined
Weight
.33 kg (.73 lb)
(3.15 cm)
(1.25")
15.25 cm
(6")
Sample
Elastomeric
Sample Holder*
7.1 cm
(2.8")
3.3 cm
(1.3")
9.4 cm
(3.7")

* Elastomeric sample holder fabricated of material firm enough to support the sample.

Figure 4. Compaction tester.

7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45-µm membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100 g minimum).

7.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Section 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples which contain oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not filter (this is usually the case with heavy oils or greases) it is carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing it to dry, weight to ±0.01 g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Section 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on depending on whether it contains more of less than 0.5% solids. If the sample appears to have less than 0.5% solids, the percent solids will be determined by the following procedure.

7.7.1 Dry the filter and residue at 80° C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids using the following equation:

$$\frac{\text{weight of filtered solid and filters} - \text{tared weight of filters}}{\text{initial weight of waste material}} \times 100 = 5\% \text{ solids}$$

NOTE: This procedure is only used to determine whether the solid must be extracted or whether it can be discarded unextracted. It is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80° C. A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid comprises less than 0.5% of the waste, discard the solid and proceed immediately to Section 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Section 7.5 and all materials that do not contain free liquids should be evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm^2 or passes through a 9.5-mm (0.375-in.) standard sieve, the operator should proceed to Section 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material would be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5-mm (0.375-in.) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Section 7.10.

7.10 Structural Integrity Procedure (SIP):

7.10.1 Cut a 3.3-cm-diameter by 7.1-cm-long cylinder from the waste material. For wastes that have been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the Extractor.

7.11 If the sample contains more than 0.5% solids, use the wet weight of the solid phase obtained in Section 7.6 for purposes of calculating the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_f - W_t$$

where:

W = wet weight in grams of solid to be charged to extractor

W_f = wet weight in grams of filtered solids and filter media

W_t = weight in grams of tared filters.

If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Section 7.11) into the extractor and add 16 times its weight of deionized water.

7.13 After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and, if the pH rises above 5.2, 0.5 N acetic acid should be added to bring the pH down to 5.0 ± 0.2. However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hr and maintained at 20°-40° C (68°-104° F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5 N acetic acid. If such a system is not available, the following manual procedure shall be employed.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2. The pH of the solution should be adjusted at 15-, 30-, and 60-min intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5 pH units.

7.13.3 The adjustment procedure should be continued for at least 6 hr.

7.13.4 If, at the end of the 24-hr extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional 4 hr, during which the pH should be adjusted at 1-hr intervals.

7.14 At the end of the extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where:

V = ml deionized water to be added

W = weight in g of solid charged to extractor

A = ml of 0.5 N acetic acid added during extraction

7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner.

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Section 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10 psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Sections 7.5 and 7.15 should be combined. This combined liquid (or the waste itself if it has less than 0.5% solids, as noted in Section 7.8) is the extract and should be analyzed for the presence of any of the contaminants specified in Table 1 using the Analytical Procedures designated in Section 7.17.

7.17 The extract will be prepared and analyzed according to the analytical methods specified in Table 1. All of these analytical methods are included in this manual. The method of standard addition will be employed for all metal analyses.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 ml of oil and 1,000 ml of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be

$$\frac{(50)(\text{contaminant conc. in oil})}{1{,}050} + \frac{(1{,}000)(\text{contaminant conc. of aqueous phase})}{1{,}050}$$

7.18 The extract concentrations are compared to the maximum contamination limits listed in Table 1. If the extract concentrations are equal to or greater than the respective values, then the waste is considered to be EP toxic.[1]

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[1] Chromium concentrations have to be interpreted differently. A waste containing chromium will be determined to be EP toxic if (1) the waste extract has an initial pH of less than 7 and contains more than 5 mg/l of hexavalent chromium in the resulting extract, or (2) the waste extract has an initial pH greater than 7 and a final pH greater than 7 and contains more than 5 mg/l of hexavalent chromium in the extract, or (3) the waste extract has an initial pH greater than 7 and a final pH less than 7 and contains more than 5 mg/l of total chromium, unless the chromium is trivalent. To determine whether the chromium is trivalent, the sample must be processed according to an alkaline digestion method (Method 3060) and analyzed for hexavalent chromium (Methods 7195, 7196, or 7197).

8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 All quality control measures suggested in the referenced analytical methods should be followed.

2.2 Mobility Procedures

Mobility procedures are used to determine the mobility of various components in a waste. Although these procedures are used to evaluate a waste, they are not to be confused with a hazardous characteristic as defined by the RCRA Regulations, Part 261.